JUNE 1952

# Chemical Engineering Progress

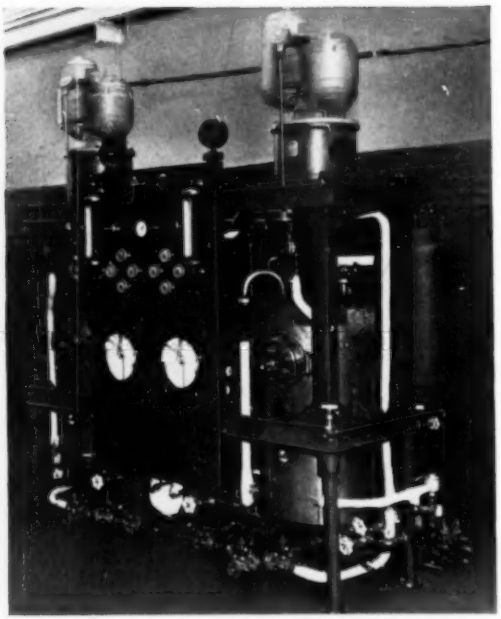
PUBLISHED MONTHLY BY THE AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

# Turbo-Topics

TURBO-MIXER
GENERAL AMERICAN
TRADE MARK

**TURBO-MIXER,** a division of
**GENERAL AMERICAN TRANSPORTATION CORPORATION**

# *MIXING*–Science and Art



TURBO-MIXER EXPERIMENTAL FERMENTERS

Assembly designed and constructed by Vulcan Engineering Division of Vulcan Copper and Supply Company.

*Chemical process mixing is a science—its methods are basically systematic and have general application. It is an art—the successful application of its methods depends to a considerable degree upon the skill and experience of the mixing engineer.*

* * *

IN THIS DEFINITION, you will find the best reasons for calling on Turbo-Mixer for counsel and aid with your mixing problems. Turbo engineers bring you almost 40 years of mixing experience — continuous background in developing the precise mixer needed for optimum results and 24 hour duty. In addition, Turbo's engineering is backed by General American's proved manufacturing ability for America's largest companies.

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# Chemical Engineering Progress

Volume 48

Number 6

JUNE 1952

CENTENNIAL OF ENGINEERING 1852 1952


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**magnetic coupling**

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***...increases Mercury Manometer accuracy***

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The F&P MERCURY MANOMETER flow meter utilizing the patented separation-proof MAGNABOND magnetic coupling brings added safety, accuracy, and maintenance-free operation to variable-head metering. The frictionless MAGNABOND coupling transmits the manometer float position without need for pressure tight bearings, lubricants, maintenance.

The F&P MERCURY MANOMETER features stainless steel elements permitting direct measurement of many corrosive fluids without the need for purges or seals. A variety of combinations of indicating, recording, integrating, and controlling Mercury Manometer instruments are available.

**WRITE FOR CATALOG 37**

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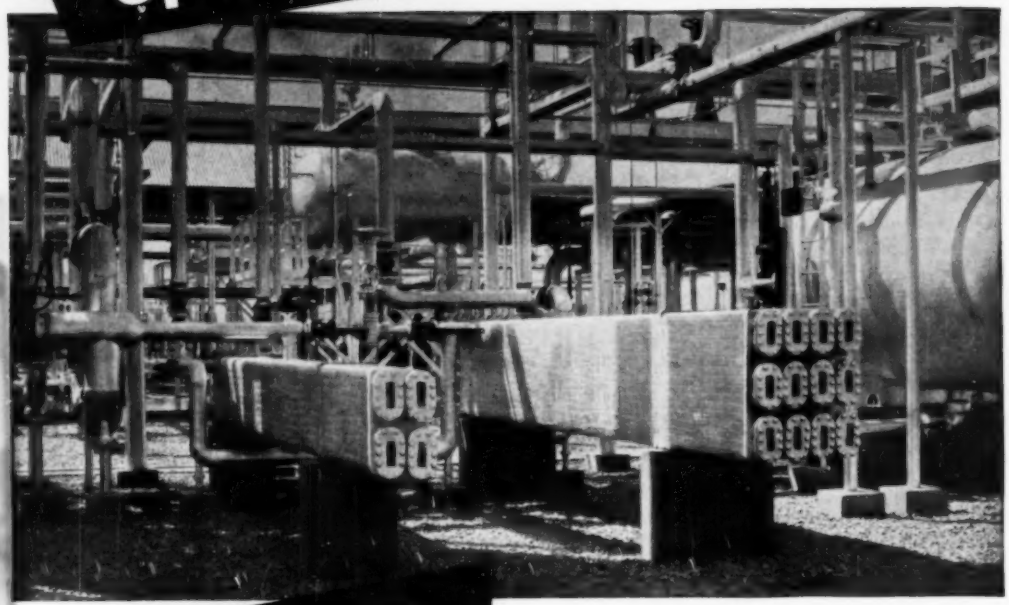
SALES ENGINEERING OFFICES THROUGHOUT THE WORLD

# Assure Continuous Operation in your plant



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● By simply manifolding an extra parallel stream into a bank of Brown Sectional Exchangers, one stream after another can be taken off-line, and cleaned, while the exchanger *operates at full capacity.*

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Sectional Hairpin Heat Exchangers
Tank Suction and Line Heaters
Fintube Heaters for Bulk Storage Tanks
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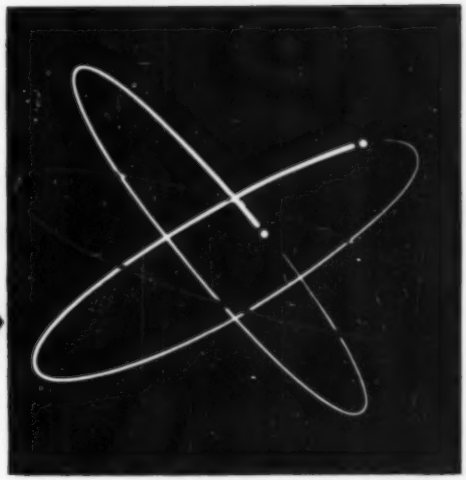
# first

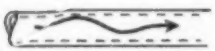
## *all-electronic control*



**of pressure**

**temperature**

**flow**



**and level**

**...**

The new Swartwout Autronic Control System is the first miniature all-electronic control system for process and power instrumentation. Completely revolutionary in conception, it is the ultimate in centralized control no matter how stringent your requirements for accuracy, speed, reliability and compactness.

Because transmission of information throughout the Autronic Control System is by electrical means *only*, there is no transmission lag; control of final element is accurate, positive and *instantaneous*. Right from the primary measuring element to the final element there is no mechanical motion whatsoever . . . no slide wires to initiate control action . . . no booster mechanisms or other delicate equipment to overcome distance effects . . . no trouble caused by air leaks or frozen air lines.

Call in our engineering representative to explain how the Swartwout Autronic Control System can bring new accuracy and speed to *your* process instrumentation and control. Or write *today* for literature on the new Swartwout Autronic Control System.

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# YOU CAN CUT CONTROL VALVE INVENTORIES *in half!*

**ONE ANNIN VALVE BODY FOR THREE TYPES OF ANNIN OPERATORS**

A single Annin valve body is standard for use with any of three types of Annin operators. Furthermore, each size of operator fits two sizes of valve bodies. Think how your inventory of critical control valves can be cut: For any installation the valve body is selected by size and alloy, then assembled with operator desired, and your control valve is complete (positioner unit built-in)—ready for service under specified conditions. In addition, all valve bodies carry a 1000# rating. Interchangeable flanges series 15, 30 and 60 are split-ring mounted to meet any requirement. Write today for detailed information. Ask for Catalog 1500B.

**ANNIN CONTROL VALVES**

THE ANNIN COMPANY

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an **economical**
stainless steel fitting
for joining pipe or tube
**without threading or welding**

# Quikup!®

Designed to reduce assembly costs and to permit the use of less expensive lighter wall tubing. ***Quikup!*** stainless steel elbows, tees, couplings, reducers and adapters save you time, labor, materials and money.

***Quikup!*** means lower installation costs! No threading, welding, flaring or other skilled assembly operations. Just cut to length and deburr.

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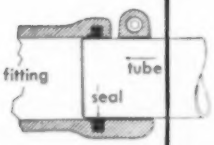
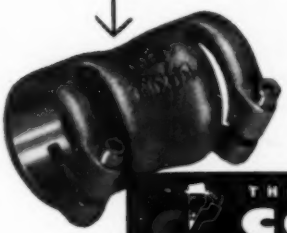
***Quikup!*** means safe, leakproof connections! Synthetic sealing rings provide positive pressure-tight joints at all times.

***Quikup!*** means simplified fitting inventories! Use it with schedules 5, 10, 40 and 80 pipe sizes without changing from one fitting to another . . . use it with a variety of tube wall thicknesses as long as the O.D. remains the same.




**TO GET THE FACTS**

about this amazing new line of stainless steel fittings, write today for Bulletin Q100



THE COOPER ALLOY FOUNDRY CO. • HILLSIDE, N. J.

*LEADING PRODUCER OF* ***STAINLESS STEEL*** *VALVES, FITTINGS & CASTINGS*

FISCHER & PORTER

# P-4

PNEUMATROL

*the 1st*

Flexible, die-cast building-block construction—six modes of control; right or left or dual case mounting, or point-of-measurement mounting.

High capacity (1.8 SCFM), non-bleed relay valve.

Double slack diaphragm design eliminates dead space and critical relay adjustment.

Large integral air passages permit maximum response, eliminate piping.

Accessible capillary restriction and integral filter.

Differential input assembly is an integral part of controller, assuring tracking.

Rugged, self-aligning "Barrel-Bearings"—eliminate friction.

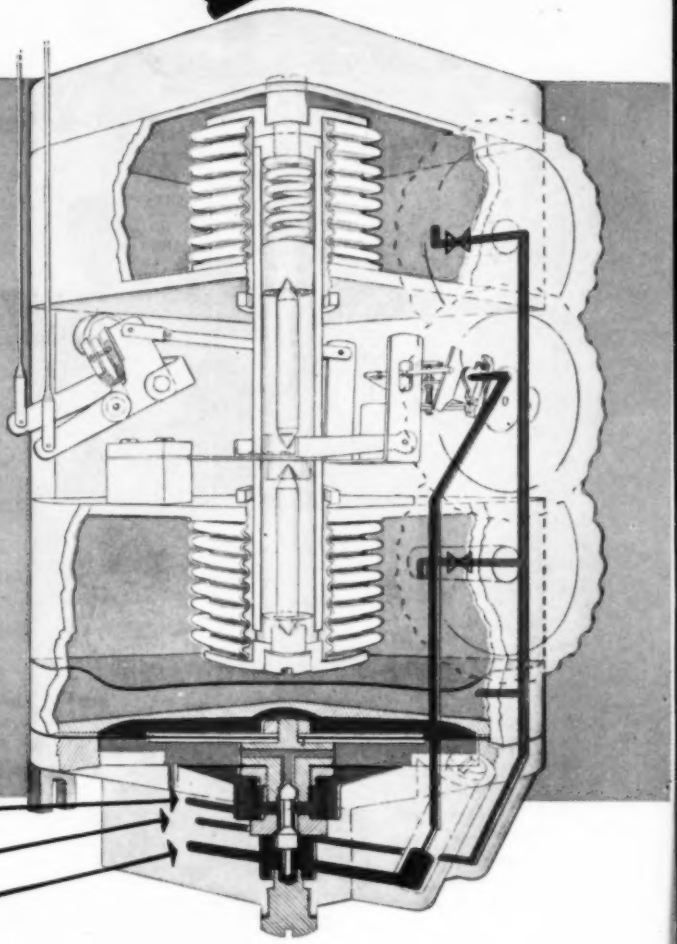
Jewel-mounted stainless steel flapper, rotatable around fixed stainless steel nozzle for proportional band adjustment and reversal of action.

Self-aligning, rigidly-coupled bellows assembly.

Beryllium-copper flexure spring, pivot-pin coupled to bellows without backlash or side thrust.

Long-path adjustable resistor valves for reset and derivative, O-ring sealed, non-jamming, calibrated 0.02—5 min. or 0.2—50 min.

Independent derivative vibration stabilizer; pneumatic balance action.

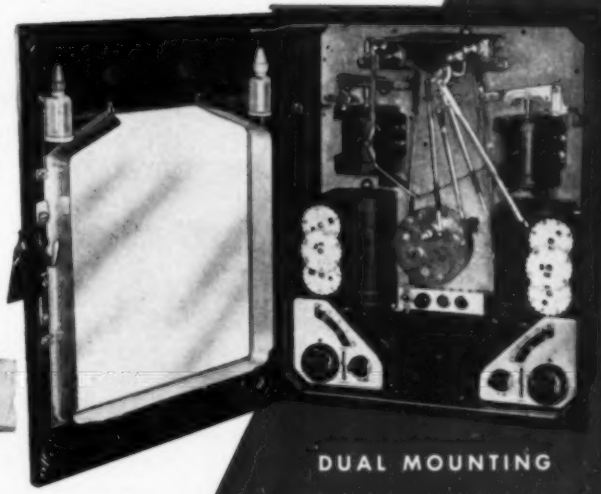


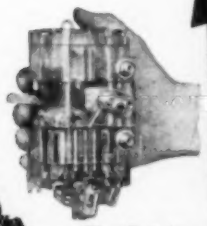
# ALL PURPOSE Pneumatic Controller

for either instrument or point-of-measurement mounting

for
- rate of flow
- pressure
- differential pressure
- temperature
- liquid level
- viscosity

The P-4 Pneumatrol is the *first* controller suitable for point-of-measurement installation as well as either single or dual case mounting. Knowing more about the many uses of the P-4 may lead to closer control of your processing operations. Get all the data NOW. The convenient coupon will bring you full information by return mail. Send for it while the subject is fresh in your mind.





The P-4 incorporates both proportional and derivative actions with automatic reset response for precise positioning... in stacked units, as shown in this transparent model.

## DUAL MOUNTING

- Complete mounting of transfer switch and regulator within the case.
- Greater panel space economy.
- New die-cast aluminum case and door.
- All accessories visible through door.
- External knobs for instant manual operation.
- *Everything* is inside the case, eliminates external manual panels.

For pneumatic-set, point-of-measurement control for use with graphic panels and miniature instruments, particularly where transmission lag must be eliminated for perfect control. Shown here mounted on the popular F & P 52 OK pneumatic transmitter.

## FISCHER & PORTER COMPANY

**160 COUNTY LINE ROAD, HATBORO, PENNA.**

Please send me by return mail complete specifications on your new P-4 Pneumatrol.

NAME_____

POSITION_____

COMPANY_____

ADDRESS_____

CITY & STATE_____

# Here's what we meant by

## Need help IN MAKING FILTERAIDS GO FARTHER?

## A survey by a Dicalite Engineer enabled this manufacturer to cut filteraid usage by 66⅔% per gallon of throughput

One of the many products in this manufacturer's line is varnish. It was being filtered by precoat only, using 100 lbs. of filteraid to deposit on the cloths of a 200-sq. ft. plate and frame press before starting to filter each 1000-gal. batch. A survey by a Dicalite engineer led to saving both time and filteraid. First, a switch was made to a higher-flowing grade of Dicalite filteraid. Only 25 lbs. of this material (Dicalite 4200) was used for the precoat, but 75 lbs. were used in continuous addition to the varnish as it was being filtered. In this way 3,000 gallons were filtered with satisfactory clarity before the press needed cleaning. RESULT: 1) 100 lbs. of Dicalite filtered 3,000 gallons instead of the 1,000 gallons put through by the former method; 2) cycle length was tripled, so that two press cleanings were eliminated in filtering 3,000 gallons and saved considerable down time and overtime. Such spectacular savings are not always possible, but our engineers find many cases where filteraid consumption could be reduced 10% to 15% without any ill effects to operation or product quality. If you feel that a check of your filtration operation or stretching your available supply of filteraid will be helpful, or if you have a current filteration problem, write our nearest office. A Dicalite engineer will gladly call at your convenience.

**Send for free copy of Bulletin B-12**

# DICALITE® FILTERAIDS

**DICALITE DIVISION, GREAT LAKES CARBON CORPORATION**

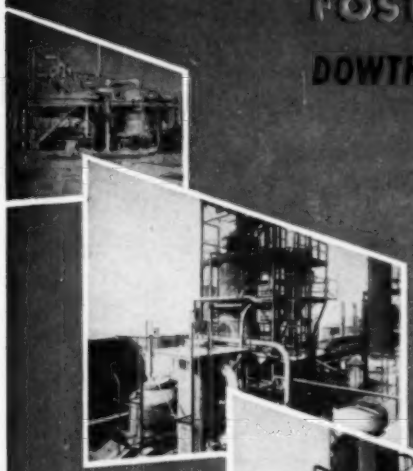
GLC

NEW YORK 17, N. Y. • CHICAGO 1, ILL. • LOS ANGELES 17, CALIF.

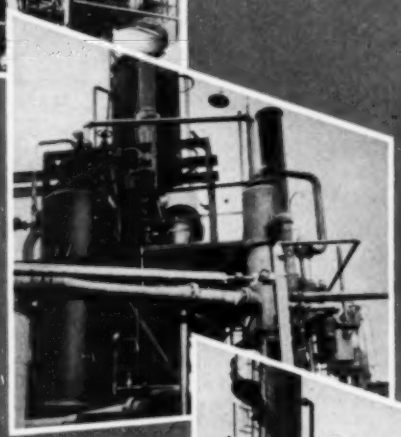
# FOSTER WHEELER

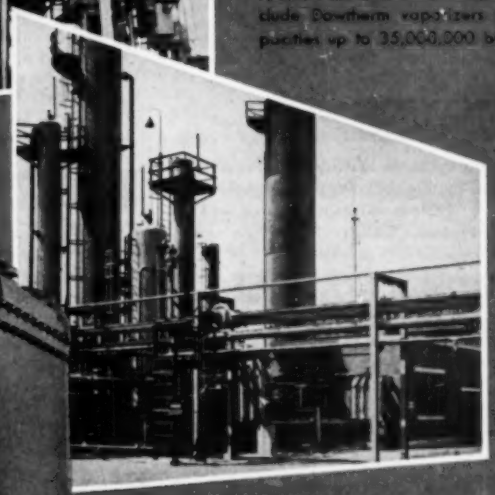
## DOWTHERM HEATED PROCESS SYSTEMS

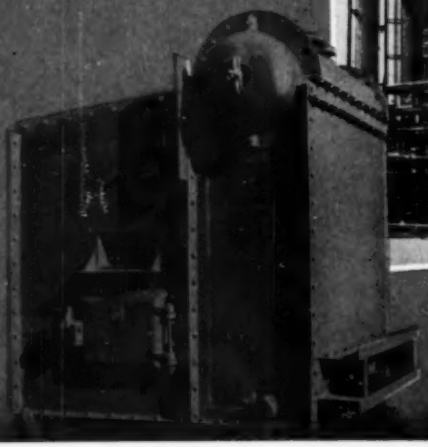
***designed, fabricated and erected under one responsibility and with one overall guarantee***



When making an installation of a Dowtherm heated process system in your plant, it is essential that all heating equipment and interconnecting piping, as well as the Dowtherm vaporizer, be properly designed and furnished by a manufacturer with sufficient background in this type of work to know the special problems involved. In 1932 Foster Wheeler Corporation, builders of heat exchangers and direct fired boilers and heaters for the past 50 years, was the first manufacturer to design, engineer, and construct a complete Dowtherm heated process system. Since that time, Foster Wheeler has installed more than 300 vapor generators and—in most cases—has supplied and engineered the entire systems. Some of these installations include Dowtherm vaporizers with capacities up to 35,000,000 btu.







...sible oil deodorization
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...sin processing
...terification
...phthalic anhydride
...stor oil dehydration
...sulphuric acid concentration
...atalyst heating and
regeneration hypersorption

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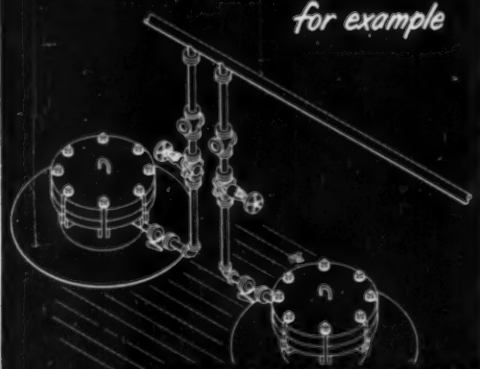
FOSTER WHEELER

# Do Your Valves Resist Corrosion Like This?

... on Digester Gases, for example



THE INSTALLATION

Crane Alloy Plug Gate Valves on sulphate digester gas-off service in a large Southern paper mill.

## VALVE SERVICE RATINGS

| | |
|---|---|
| CORROSION-RESISTANCE: | Best results ever had |
| FEATURES: | Plug seating-good for throttling and wide open service |
| SUITABILITY: | Adopted as standard |
| MAINTENANCE COST: | No upkeep needed |
| SERVICE LIFE: | Hardly any wear after 2 years |
| OPERATING RESULTS: | Better control of digester gases |
| AVAILABILITY: | Regular Crane Catalog item |

## THE HISTORY

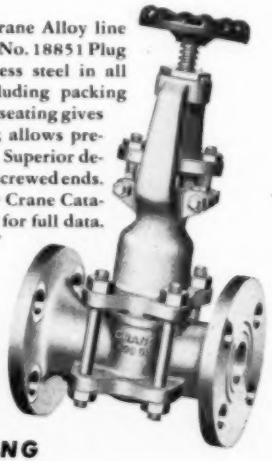
Valves in this installation are used to relieve, under throttled flow, the highly corrosive sulphate gases from digesters. Various valves tried gave constant trouble with corrosion, leakage, and difficult operation. Replacements were excessive.

Then Crane 18-8 Mo Alloy Plug Gate Valves were installed on a test basis. After 1 year's service, and again after 2, careful inspection showed hardly a sign of wear or corrosive effects. Meanwhile, the valves operated smoothly; required no maintenance.

So well have Crane valves out-performed and outlasted all others tried, they have been made standard equipment on all digesters.

## THE VALVE

One of the leaders in the Crane Alloy line for severe corrosive service—No. 18851 Plug Gate—with 18-8 Mo stainless steel in all parts exposed to flow, including packing gland. Circular, tapered plug seating gives minimum resistance to flow; allows precise control when throttling. Superior design throughout. Flanged or screwed ends. Also in All-Monel. See your Crane Catalog or Crane Representative for full data.



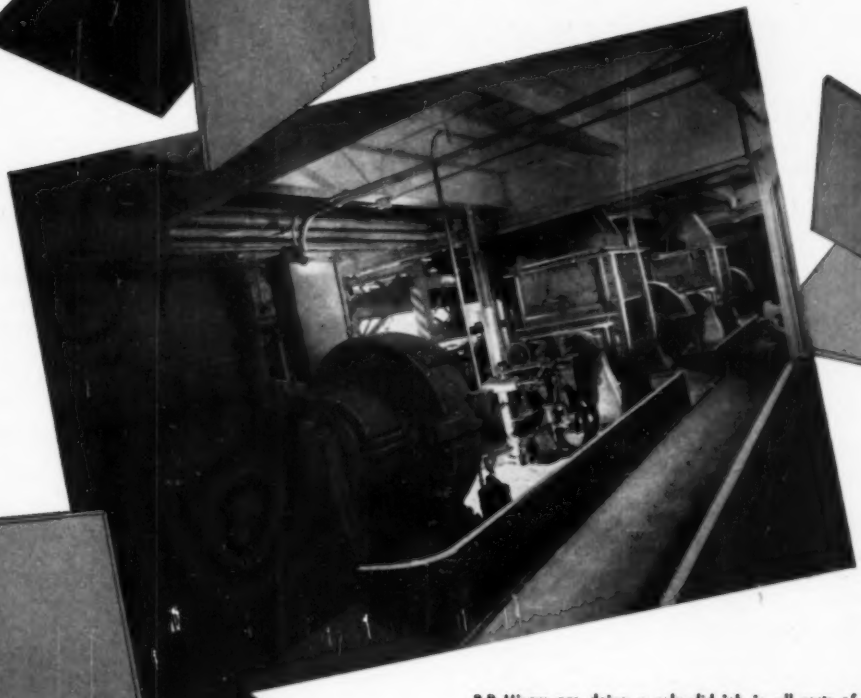
*The Complete Crane Line Meets All Valve Needs. That's Why*
**More Crane Valves Are Used Than Any Other Make!**

# CRANE VALVES

CRANE CO., *General Offices:* 836 S. Michigan Ave., Chicago 5, Illinois
*Branches and Wholesalers Serving All Industrial Areas*

***VALVES • FITTINGS • PIPE • PLUMBING • HEATING***

# Colorful asphalt floor tile mix uniformly produced in BAKER PERKINS mixers



B-P Mixers are doing a splendid job in all sorts of processing operations, and this installation of six size 15JSE BAKER PERKINS Mixers at a large asphalt floor tile plant is an excellent example. These six mixers can thoroughly blend the ingredients for asphalt floor tile at the rate of 18,000 lbs. per hour on a continuous production schedule. Like other BAKER PERKINS equipment, the cost of operating and maintenance, including labor, material handling, power, and depreciation, are down where they should be. BAKER PERKINS equipment can help cut costs, increase production, and improve the quality of your product, too. Consult a B-P sales engineer or write us today.

**BAKER PERKINS INC.**
CHEMICAL MACHINERY DIVISION
SAGINAW, MICHIGAN

# Applications Unlimited...

*wherever you need to measure,*

*. . . choose*

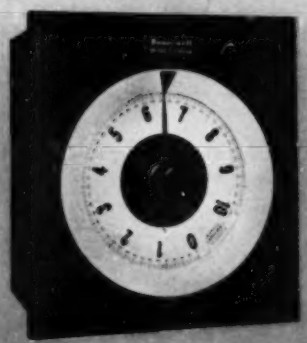
*ElectroniK*
*Potentiometers*

WHETHER charting the course of an experiment on a laboratory bench, or regulating huge-scale production in a sprawling processing plant, *ElectroniK* Potentiometers point the way to accelerated research, to greater productivity, to higher quality, and to lower costs.

Versatile *ElectroniK* Instruments are supplied calibrated for variables such as pressure, temperature, level, flow, pH, conductivity, speed and motion. A wide selection of instruments provides a choice of indicators, single and multi-point recorders, and a variety of electric and pneumatic

**PRECISION INDICATOR**

centralizes up to 48 different readings on one instrument; just press a button and wide-open scale turns quickly to measured value.



**INDICATING CONTROLLER**

set point and measured values are easily read on 2 ½-foot circular scale. Supplied for on-off and proportional electrical control.

● *Important Reference Data...*

# *record or control*

controllers from the simplest to the most complex types. In all these, the *ElectroniK* "continuous balance" measuring system, with electronic non-cyclic balancing, affords the peak of speed, sensitivity and service-proved dependability.

Plan now to gain the full benefits of automatic instrumentation throughout your plant. Call in your local Honeywell engineer . . . he is as near as your phone.

MINNEAPOLIS-HONEYWELL REGULATOR CO., *Industrial Division*, 4427 Wayne Avenue, Philadelphia 44, Penna.

## Of special interest to research men . . .

### FUNCTION PLOTTER

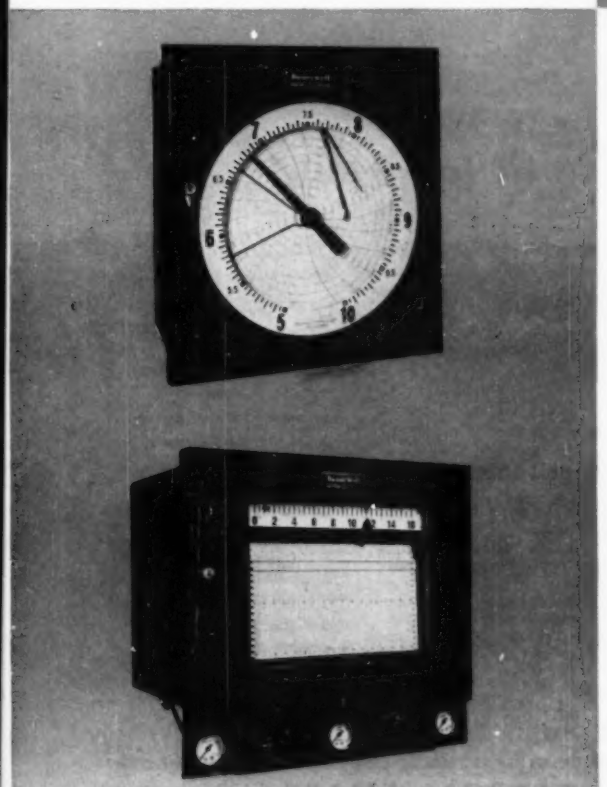
automatically charts temperature vs. expansion, speed vs. torque, stress vs. strain. One variable actuates the pen, the other actuates the chart drive.

### DUPLEX RECORDER

simultaneously records any two independent variables. Two pens actuated by separate measuring systems can both travel full width of the chart.

### ELECTROMETER

records currents as small as $10^{-15}$ amperes, for measurements with ionization chambers, spectroscopes, etc.; features fast response, low noise, high stability.



### Recorders

Circular chart type with 28⅝" scale can be read at a distance. Strip chart type records as many as 16 points. Scanners available for larger number of points. Pen speeds as fast as one second.

### Electric Controllers

Circular chart and strip chart types are supplied for on-off and proportional control . . . Electr-O-Line for position-proportioning, Electr-O-Pulse for time-proportioning.

### Pneumatic Controllers

Choice includes on-off control, Limited Throttlor and Full Throttlor controllers with manual reset, Air-O-Line Controller with automatic reset. Rate action is optional.

MINNEAPOLIS **Honeywell**

BROWN INSTRUMENTS

*First in Controls*

**For a brief description of all Honeywell instruments, write for Composite Catalog 5000.**

*How Celite Mineral Fillers give a product better dispersion . . .*

# Making a better "killer" of insecticides

To increase the effectiveness of their product... add more "killing power"—many leading producers of insecticides add Celite Mineral Fillers to their dust as a standard ingredient.

This use of these diatomite powders is based primarily on (1) their light weight and great bulk which improve dispersion of the poison, and (2) their high absorption capacity that produces dry dust concentrates from both low melting point solids and liquid poisons, thereby increasing the potency of the final product.

These and other unusual physical characteristics adapt Celite Mineral Fillers to numerous industrial uses.

**CHECK LIST OF PRODUCT BENEFITS OBTAINABLE AT LITTLE COST WITH CELITE MINERAL FILLERS**

- Added Bulk
- Better Suspension
- Faster Cleaning Action
- Greater Absorption
- Improved Color
- Better Dielectric Properties
- More Durable Finish
- Increased Viscosity
- Elimination of Caking
- Higher Melting Point
- Better Dry Mixing
- Improved Dispersion

**THESE CELITE PROPERTIES BENEFIT MANY TYPES OF PRODUCTS**

Because of their inertness and great bulk per unit of weight, Celite Mineral Fillers make ideal bulking agents for powders and pastes. Their tiny multi-shaped particles interlace to stiffen and strengthen admixtures. The microscopically small facets of these particles diffuse light so effectively that they will give any desired degree of flatness to a surface film. And their porous, thin-walled cellular structure can be utilized to impart a delicate, non-scratching abrasive action.

You may find Celite the "extra something" needed to lift your product above competition. Why not discuss its application to your problem with a Celite engineer? For further information and samples, write Johns-Manville, Box 60, New York 16, N. Y. In Canada, 199 Bay St., Toronto, Ontario.

JOHNS MANVILLE JM PRODUCTS

**Johns-Manville CELITE®**

**MINERAL FILLERS**

For 25 years McKesson and Robbins' formula for Glycerine suppositories has remained unchanged. Public confidence in Glycerine as an effective ingredient has been thoroughly demonstrated by the wide acceptance of this product as a familiar "medicine cabinet" accessory.

**FULLY ACCEPTED... FULLY AVAILABLE IN ALL GRADES!**

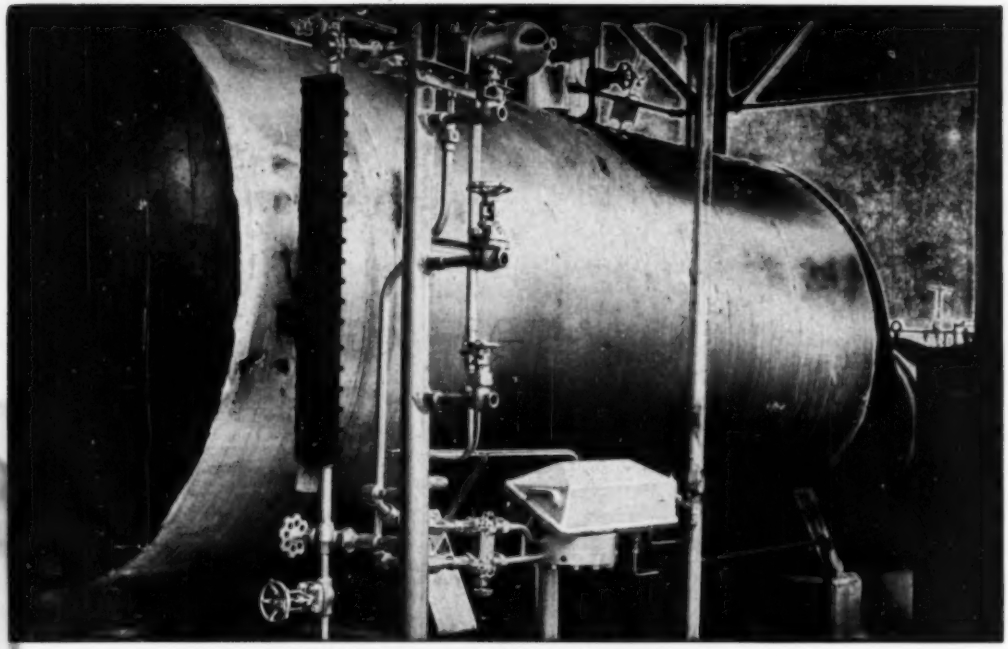
U.S.P. Glycerine—in children's and adult suppositories—has long been an indispensable ingredient among drug manufacturers . . . primarily for its mild stimulating and lubricating qualities.

But versatile Glycerine often contributes a great many more advantages to jelly- or emulsion-based preparations of this type. For instance, its hygroscopic action keeps the mixture from drying out. It's a natural plasticizer. And in salves for topical use, U.S.P. Glycerine acts as an ideal solvent for the medicament.

Hundreds of applications in a host of proprietary pharmaceuticals attest to the fact that Glycerine has won the trust of American consumers over the years . . . that its presence on your label is accepted instantly . . . that it is widely recognized as a product which can be safely applied internally or externally. We've outlined some of these applications in a booklet, "Why Glycerine for Drugs and Cosmetics?" Write for your copy—today!

**GLYCERINE PRODUCERS' ASSOCIATION**

295 Madison Avenue, New York 17, N. Y.



Republic Transmitter measuring level in reboiler

# DIFFERENT . . . in design and performance

The Republic Pneumatic Transmitter is a device for converting process variables, such as flow, level, pressure or liquid density, into air pressures. These air pressures are a direct measure of the process variables or can be used as the measuring impulse for the actuation of an automatic controller.

The force-balance method of measurement, as employed by the Republic Pneumatic Transmitter, offers many inherent advantages such as:

**AMBIENT TEMPERATURE** — The effect of ambient temperature variations on the accuracy of a Republic Transmitter is negligible. Since all parts are equally affected by temperature changes, force and leverage relationships are not disturbed and accuracy is unimpaired.

**AIR SUPPLY PRESSURE** — The effects of changes in air supply pressure is so small that it is guaranteed negligible.

**LINE PRESSURE** — The effect of variations in line pressure has been completely eliminated.

**SENSITIVITY** — Due to the negligible motions required for complete operation of all parts for full scale changes, no appreciable hysteresis results from reversal of direction of measurement change. The hysteresis loop is so small that it is undetectable by ordinary means, being less than 1/20 of 1%.

**ACCURACY** — The accuracy of the Republic force-balance method of measurement is higher than can be consistently secured and maintained with any other method. Transmitting pressure vs. measured force is guaranteed within ½ of 1% of meter range.

In addition to these five important features, the Republic Pneumatic Transmitter is extremely flexible in application, easy to service and of rugged construction.

If you have a flow, level, pressure or liquid density metering or control problem may we suggest that you investigate the Republic Pneumatic Transmitter? Data Book No. 1002, which contains complete details of this instrument, will be mailed to you upon request. Write for it today!

**REPUBLIC FLOW METERS CO.**

2240 Diversey Parkway, Chicago 47, Illinois

**After two years of development and test work confirmed in the operation of the large West Texas installation pictured below,**

# HUDSON presents the

# Combin-aire*

**A New Type Cooling System Integrated for All Process Plant Cooling Services, Combining the Economical Maximum Use of Air With Minimum Use of Water.**

In the COMBIN-AIRE the use of air for cooling is extended to include low temperature cooling services previously necessitating water cooling because of high ambient air temperature. The COMBIN-AIRE allows the use of air-cooling for total heat dissipation in all services.

The COMBIN-AIRE is a combination of a HUDSON water cooling tower with HUDSON air-cooled exchangers housed in one integrated structure. During periods of high ambient temperature the inlet air is first drawn through water at the bottom of the cooling tower, reducing the air to near wet bulb temperature, at which temperature it contacts the air-cooled exchangers. During periods of low ambient temperature the inlet air is used directly in the air-cooled exchangers and no water is evaporated in the cooling tower.

When water in good quantity and quality is not available the COMBIN-AIRE has manifest advantages over any alternative combination of cooling equipment.

Even when ample water is available and water treatment costs are low, comprehensive comparisons will frequently indicate overall advantages of the COMBIN-AIRE system.

*** The COMBIN-AIRE unit is protected by United States patents**

**COMBIN-AIRE ADVANTAGES**

- **LOW WATER CONSUMPTION.** No water is used during cold weather.
- **NO TREATMENT NECESSARY** for cooling tower water.
- **NO WATER LOSS** as spray or mechanical carryover.
- **NO CONDENSATION** from effluent air.
- **MINIMUM PIPING.** COMBIN-AIRE may be installed immediately adjacent to other process equipment. Use of tubular water cooled equipment for final coolers unnecessary.
- **CLEAN AIR TO AIR-COOLED UNITS.** During hot season air is water-washed removing dust, sand and insects.

**HUDSON**
**ENGINEERING CORPORATION**
**FAIRVIEW STATION • HOUSTON, TEXAS**

This COMBIN-AIRE unit operated by the [illegible] Oil Company at [illegible] cools [illegible] from oil absorption and fractionating plant streams, a total heat load of about [illegible]

# HUDSON

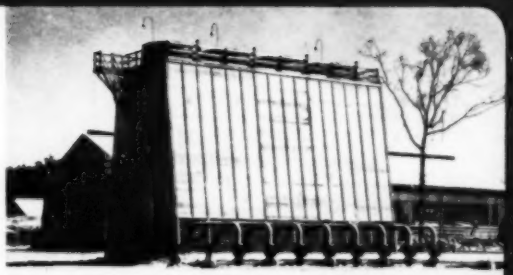
Top Performance

# COOLING EQUIPMENT

*For more than two decades HUDSON personnel have developed equipment for heat recovery and heat dissipation. Present day HUDSON cooling equipment has been evolved through the years by constant improvements in design and fabrication.*

## WATER COOLING TOWERS

Beauty and utility have been successfully combined in the HUDSON "TP" design. These Top Performance cooling towers embodying new but proven advancements in water cooling design, result in lower installation and operating costs for a given guaranteed performance.

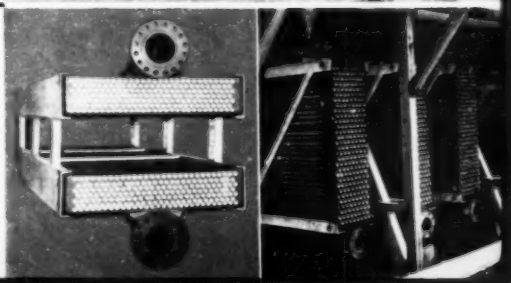


## SOLO-AIRE UNITS

HUDSON SOLO-AIRE UNITS for jacket water cooling, gas cooling, steam condensing and hydrocarbon vapor condensing have found wide use, particularly where suitable cooling water is impossible or expensive to obtain. Even where adequate quality and quantity of water is available there are conditions under which these units have marked advantages over alternative cooling methods. Designs are available for pressures up to 5,000 pounds per square inch.

## ATMOSPHERIC SECTIONS

These units are for use where water serves as cooling medium. They may be mounted vertically or horizontally, under cooling tower or submerged in water.



**UNDIVIDED RESPONSIBILITY** — Complete integration of facilities in selection, design and manufacture of cooling towers, water cooled atmospheric condensers, and air cooled exchangers results in cooling systems of minimum first cost, minimum operating cost, and undivided responsibility.

**HUDSON**
**ENGINEERING CORPORATION**
FAIRVIEW STATION • HOUSTON, TEXAS

Petroleum refining and chemical manufacturing are industries of almost incredible superlatives. They have spent for new facilities since 1945 a sum roughly equal to total gross plant investment for all prior years. In these accomplishments, The Lummus Company—as design engineers and constructors—was privileged to play a major role.

But the years ahead are apt to make the past look puny by comparison. This year, for example, oil companies will top 1951 capital spending by over a third—chemical companies by nearly a fifth. Preliminary plans through 1955 show little or no relaxation from this rate.

Big job? Lummus is ready—a fact you may wish to bear in mind as mounting demands challenge the already-extended organizations of petroleum and chemical companies.

Rely on the quality and versatility of the Lummus staff—on the proven ability of Lummus to handle, under one contract, the functions of engineering, drafting, purchasing, expediting, inspecting, erection and initial operation.

And—as a policy statement for the record—rely on us to recognize and accept the special responsibility of handling confidential information in accordance with your own best interests and desires.

May we work together? We're ready!

**THE LUMMUS COMPANY**

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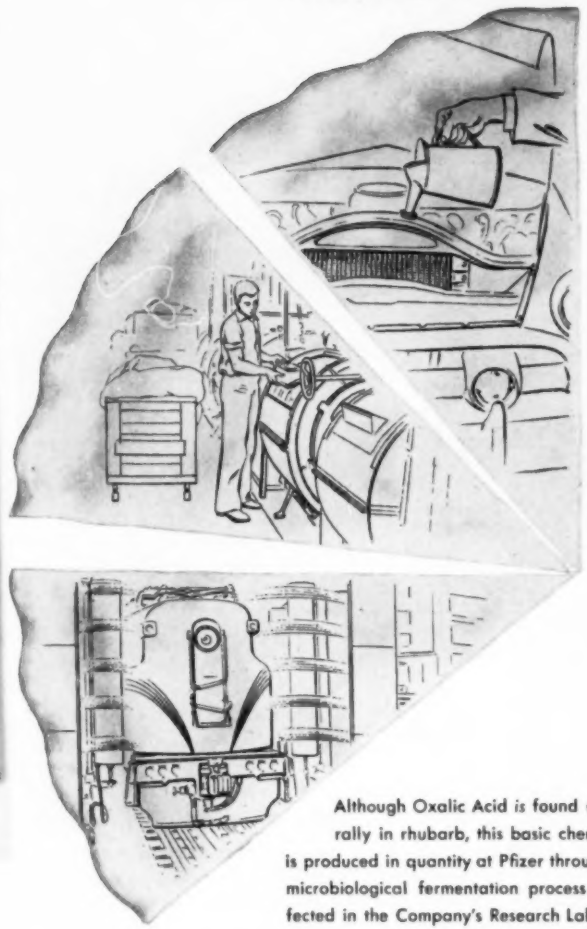
DESIGNING ENGINEERS AND CONSTRUCTORS FOR THE PETROLEUM AND CHEMICAL INDUSTRIES

# Rhubarb's Fine for Pies...



**but it won't fill your needs for**

# Oxalic Acid

Although Oxalic Acid *is* found naturally in rhubarb, this basic chemical is produced in quantity at Pfizer through a microbiological fermentation process perfected in the Company's Research Laboratories. The Pfizer process yields Oxalic Acid of high purity.

Because of its ability to solubilize iron oxide, Oxalic Acid is highly effective as an ingredient of metal cleaning compounds, in radiator flushes and as a rust remover in textile and leather processing. Its bleaching properties make it useful as a laundry sour and in the treatment of straw, cork and leather.

For more detailed information on the varied uses of this important organic acid, write for Technical Bulletin #34.

**CHAS. PFIZER & CO., INC.**
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*Manufacturing Chemists For Over 100 Years*

PFIZER QUALITY

PFIZER

# Only PYREX® CASCADE COOLERS

## give you double protection against corrosion

**1** Stop chemical attack inside tubes

**2** Permit use of low cost water as coolant

You're doubly protected against corrosion—doubly assured of processing economies with PYREX brand Cascade Coolers. First, you can count on long service life with low replacement costs because of their exceptional resistance to practically all liquids. Second, you can use low cost sea or river water as a coolant. Then, too, PYREX Cascade Coolers offer you:

**DURABILITY**—They withstand rapid temperature changes and mechanical shock and the corrosive action of chemicals.

**ASSURED PRODUCT PURITY**—They provide positive protection for CP chemicals, drugs, foods and other products susceptible to contamination.

**FURTHER ECONOMY**—Low in first cost per BTU transferred, PYREX brand Cascade Coolers have "glass-smooth" surfaces which resist scale formation—keep efficiency up . . . cleaning and maintenance costs down.

**HIGH VERSATILITY**—You can mount them on floor, wall or ceiling to conserve space—use them in series or parallel. Shipped complete, an eight-tube unit requires only a few hours for assembly by your own men.

**Handy nomographs and tables to help you compute your heat transfer requirements, along with a complete description of PYREX Cascade Coolers are included in Corning's new Technical Bulletin PE-8. Send for your copy today. Mail the coupon below.**

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Please send me a copy of Technical Bulletin PE-8, "PYREX Cascade Coolers" and name of nearest distributor.

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# HOW MUCH DO FILTER

Have you ever thoroughly investigated both the direct and indirect cost of filter cloths in your plant? If not, the chances are you will be surprised to find that annual cloth replacement and maintenance cost alone runs from $1.00 to $4.00 per square foot of press area per year, depending on the utilization of your press, the product and the type of fabric you use.

Other costs associated with use of cloths, such as the labor for scraping, handling, washing; breaking and redressing the press; and purchasing time expended in obtaining the cloth you need when you need it will probably be even greater, even if not as easily pinned down.

Here then are three immediate reasons why you should consider switching to modern high-speed Niagara pressure leaf filters:

**1. Niagara filters require no filter cloths.** Accurate case histories in our files show that some plants amortize their Niagara filters in a few years on this saving alone.

**2. Niagara filters require less labor.** When you eliminate filter cloths, you eliminate the back-breaking and costly labor that goes with them. With Niagaras you get a quick, easy, clean operation that takes only a few minutes for one man between cycles.

**3. Niagara filters work harder for you.** Reduced downtime means more production time and therefore higher production at your filter station. The filter works for you more hours per day—gives you many more gallons throughput with less installed filter area.

●

By switching to Niagaras, many press users have realized over-all savings that amortize the cost of the new filters in months.

Entirely aside from savings, here are nine sound reasons for installing Niagara Filters:

**1. Higher flow rates** on an equal area basis. Niagaras normally give flow rates 2 to 5 times those of conventional cloth-covered filter presses. This is because the rigid metal screens do not "give" under pressure—cannot obstruct flow channels in the drainage member as does sagging fabric cloth.

**2. Sparkling filtrate clarity.** Positive removal of solids to practically any degree of clarity you desire. In sanitary applications, 0-0 bacteria count can usually be secured in one filtration.

**3. No leakage.** Fully enclosed, pressure-tight construction eliminates drippage and fumes. Ideal for handling flammable, explosive, toxic, volatile, or highly obnoxious liquors.

**4. Excellent cake-washing characteristics.** Nearly true displacement washing is obtainable. Wash liquid is a minimum.

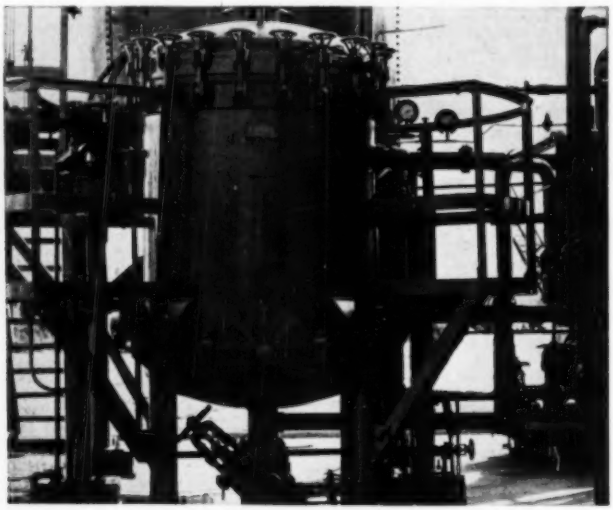
**5. One-man operation and cleaning.** Single or multiple units can be operated and cleaned easily by one man. The entire leaf battery is accessible for easy cleaning.

**6. Corrosion resistance.** The pressure leaf filter can more cheaply and readily be constructed of stainless steel, nickel, monel and other corrosion-resistant materials.

**7. Jacketed construction.** Niagaras can readily be supplied with steam jackets or special insulation.

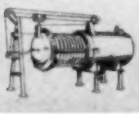
**8. Wide capacity range.** Sizes range from 20 to 950 square feet of filter area, with average capacity of 10 to 425 G.P.M. in one compact unit. (Even larger areas are available in the new horizontal filter shown at right on the opposite page.)

**9. Lower maintenance cost.** There are no moving parts during filtration. Simple construction, with fewer parts to wear out, means low-cost upkeep.



THIS NIAGARA FILTER, in large petrochemical plant, removes iron sulfide particles from solutizer solution.


Niagara Filter CORPORATION



HORIZONTAL for non-aqueous liquids; high solids; cake recovery.

IN EUROPE—NIAGARA FILTERS EUROPE, 36 Leidsegracht, Amsterdam-C, Holland

# CLOTHS COST YOU?

## NEW NIAGARA STYLE H* FILTER

### Low cleaning time . . . no cloth cost

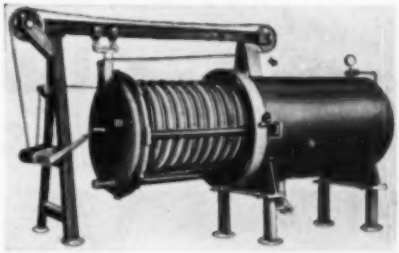
This new Niagara filter answers the need for a filter which can quickly discharge large quantities of semi-dry cakes. A variation of the standard Niagara design, it still retains all the basic advantages of Niagara vertical pressure leaf filters.

A retractable carriage permits all leaves to be withdrawn at once for rapid cleaning. One man can easily discharge as much as 150 cu. ft. of semi-dry cake in a matter of minutes merely by rapping the leaves.

A new closure design, the Q/O* (quick-opening) cover, permits faster, easier opening and closing than ever before in a filter of this size.

The Style H filter, available in sizes up to 1500 sq. ft., is ideal for filtration where high percentages of solids must be removed or where solids must be recovered. It is also recommended for standard liquid clarification where low headroom or other special reasons make its horizontal construction preferable to a vertical filter.

*Trade Mark Patent Applied For.



## HOW THE NIAGARA FILTER WORKS

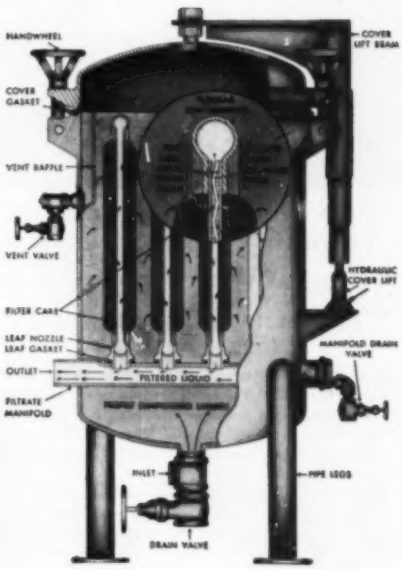
The leaves, which are the heart of the filter, consist of double-faced screen assemblies bounded by a tubular drainage frame leading to an outlet nozzle.

The unfiltered liquid usually with filteraid in suspension is pumped into the closed pressure filter tank in which the leaves are placed, and is forced through the fine-mesh screens, on which the filter cake is formed by the solids in the liquid.

Only the clear liquid reaches the inside of the leaves, flowing through the coarse center drainage wire to the tubular frames, and out through the leaf nozzles to the manifold.

When the batch has been completely filtered, or when the cake has nearly filled the space between the leaves, the filter is emptied and the filter cake is sluiced from the leaves with a hose or with automatic high-pressure sprays. Sluicing is done without removing any parts from the filter. (Cake removal for the Style H filter is somewhat different, as described above.)

As a rule, the filter is ready to go to work again in 15 minutes to one hour, depending on its size.




### THESE 5 SERVICES HELP MAKE HIGH-SPEED, LOW-COST FILTRATION A REALITY IN YOUR PLANT:

1. **Nationwide field service.** Niagara representatives in 24 cities know filtration and can give you facts.

2. **Modern filtration laboratory** determines your requirements accurately by test-filtering samples of your materials.

3. **Pilot filter rental** plan enables you to "preview" Niagara performance in your plant, at low cost.

4. **Custom engineering.** A single filter or a complete system, designed for your needs and built in materials of your choice.

5. **Installation and start-up** are supervised by Niagara factory engineers, always on call.

PILOT FILTER duplicates operation of large production models.

Operating figures prove the economic soundness of replacing cloth-covered presses with Niagara filters. For details, write us today. Or mail the coupon for descriptive literature.

**STANDARD VERTICAL** —for clarification and "polishing".

**"AUTO-SLUICE"** for fully automatic cake sluicing.

**NIAGARA FILTER CORP., 3091 Main St., Buffalo 14, N. Y.**

Please send: ☐ General literature; ☐ Data on horizontal filters; ☐ Data on pilot filter rental

Name_____

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Company_____

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# *Simplest Flow Measurement*

## with the ALL-METAL Foxboro d/p Cell!

**. . . gives fastest response, longest sustained accuracy, lowest installation cost**

Simpler in design, simpler and much less expensive to install, far easier to maintain . . . the all metal Foxboro mercury-less d/p *Cell* offers greater speed of response, permitting closer flow *control*, than has ever been possible before. Its negligible displacement and corrosion-proof construction of Type 316 Stainless Steel eliminate usual maintenance problems, even in service on highly corrosive or viscous fluids previously considered unmeasurable.

The Foxboro d/p Cell measures differential pressure by the force-balance principle and transmits pneumatically to indicating, recording, or controlling receivers. Small, compact, weighs as little as 19 lb. Ranges: from 25" to 800" $H_2O$. Working pressure ratings up to 4000 psi. Steel or stainless steel construction. Inherent over-range protection. Easy in-the-field calibration and range changes. Optionally available with pre-assembled manifold piping shown in photo above.

Thousands of d/p Cells now in use throughout industry, with many repeat orders now on our books, indicate the wide acceptance and successful performance of this revolutionary development for the measurement of liquid, steam, gas, or air flow. Write for detailed Bulletin 420. **The Foxboro Company, 936 Neponset Avenue, Foxboro, Massachusetts, U. S. A.**

- No seal pots
- No mercury
- No leveling
- Installed at the orifice

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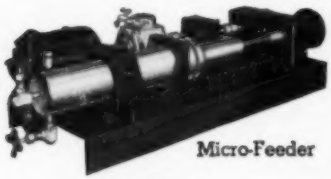
# CHEMICAL PROPORTIONING PUMPS

## *for Every Process Plant!*

**FROM PILOT PLANT OR LABORATORY USE TO THE LARGEST INSTALLATION . . . . .**

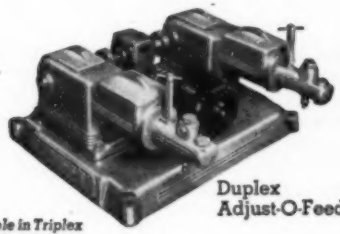
### PLUNGER TYPE PUMPS

FOR HIGH PRESSURES



Micro-Feeder

Simplex
Adjust-O-Feeder*

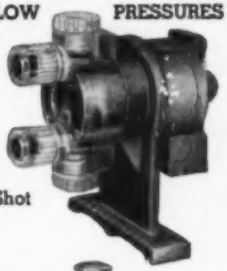


Duplex
Adjust-O-Feeder

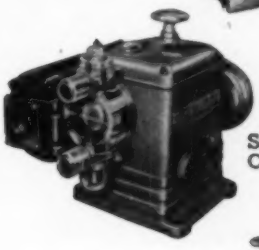
*Also available in Triplex and Quadruplex designs

### DIAPHRAGM TYPE PUMPS

FOR UNIVERSAL APPLICATION AT LOW PRESSURES

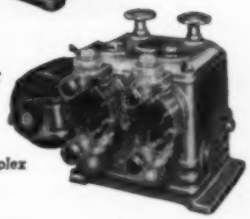


Micro-Shot



Simplex
Chem-O-Feeder*

Duplex
Chem-O-Feeder



*Also available in Triplex

### COMPLETE FLEXIBILITY OF APPLICATION

%Proportioneers, Inc.% can supply proportioning pumps, with control panels, chemical tanks, dissolvers, and all other units required — a single responsibility for your complete chemical feeding system. Consult us regarding the layout and installation of chemical proportioning equipment for automatically treating, blending, diluting and sampling in process operations.

*Write for Bulletins.*

## % PROPORTIONEERS, INC. %

Write to %PROPORTIONEERS, INC.%, 419 Harris Ave., Providence 1, R. I.

Technical service representatives in principal cities of the United States, Canada, Mexico and other foreign countries.

**RAW MATERIAL** shortage will never hit the Northwest Magnesite Company's Cape May, New Jersey plant, for its basic source of supply is seawater—its basic end product, magnesia. In this utilization of modern processing techniques to tap the virtually unlimited chemical resources of the sea, sedimentation is of primary importance.

At Cape May, the jobs of preliminary treatment of seawater and subsequent thickening and washing of magnesium hydrate are carried out in Dorr Thickeners—six of them, ranging from 150 to 320 feet in diameter.

It is particularly significant that these steps are handled by Dorrs at every major seawater magnesia operation in the U. S. . . . regardless of whether the end product is refractory, metal or both.

No matter what the application, when you need thickeners it will pay to check Dorr . . . the oldest manufacturer of sedimentation equipment *with the newest ideas.*

**BETTER TOOLS** *today* **TO MEET TOMORROW'S DEMAND**

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# A Headline that is also a Helpline!

**1. BUFLOVAK CHEMICAL AND FOOD PRODUCT EVAPORATORS**

**2. PROFITABLY MEET YOUR EXACTING REQUIREMENTS**

**3. BY DOING THE JOB BETTER AT LOWER COST**

**4. AND THE RESULT FOR YOU — GREATER PROFITS**

1. For concentrating Chemicals, Pharmaceuticals, Antibiotics, and Food Products, BUFLOVAK Evaporators offer worthwhile operating advantages.

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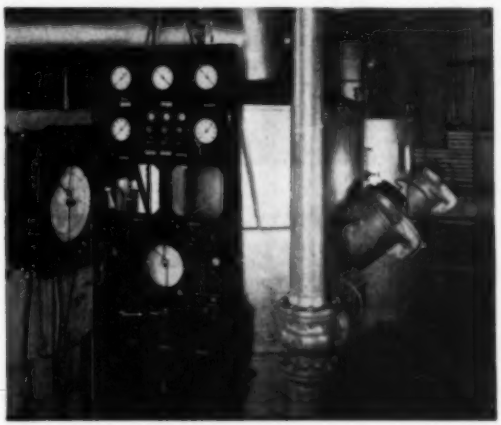

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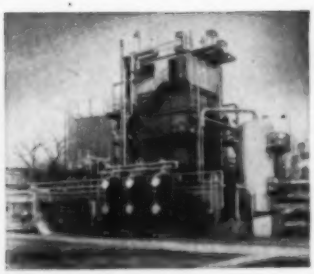

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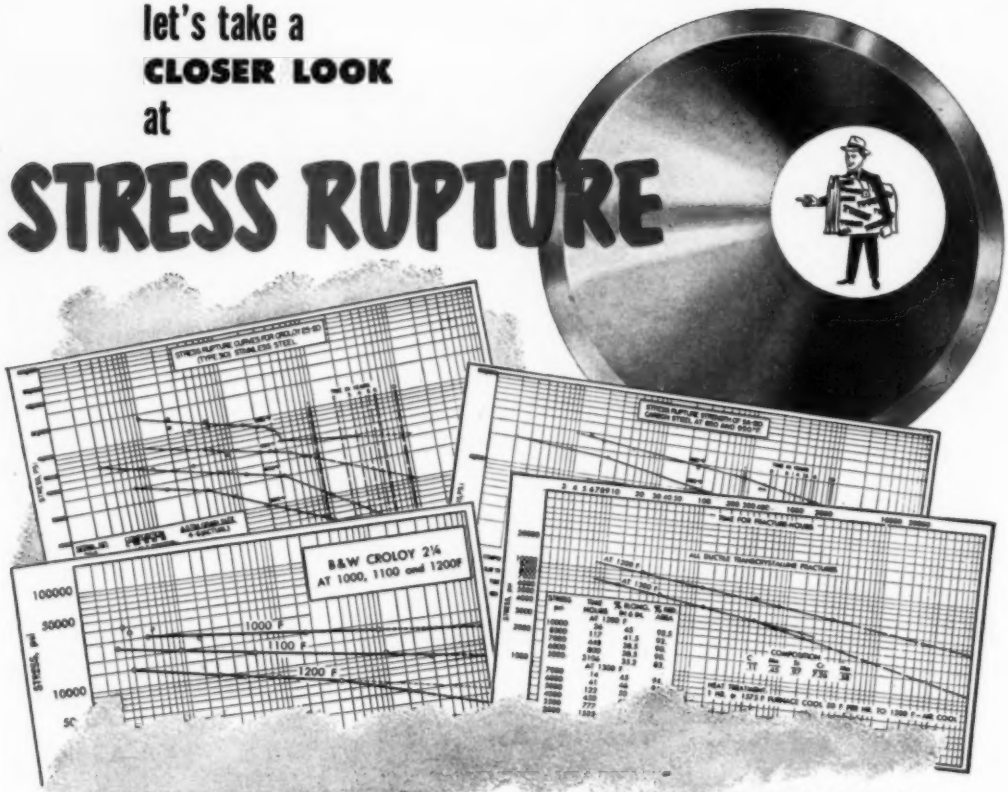
# let's take a CLOSER LOOK at STRESS RUPTURE

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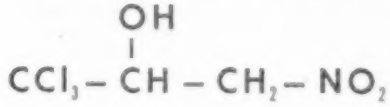

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| **Boiling point (approx.), 5 mm Hg** | **120°C** |
| **Melting point** | **40°C min.** |
| **Specific gravity, 45/4°C** | **1.605** |

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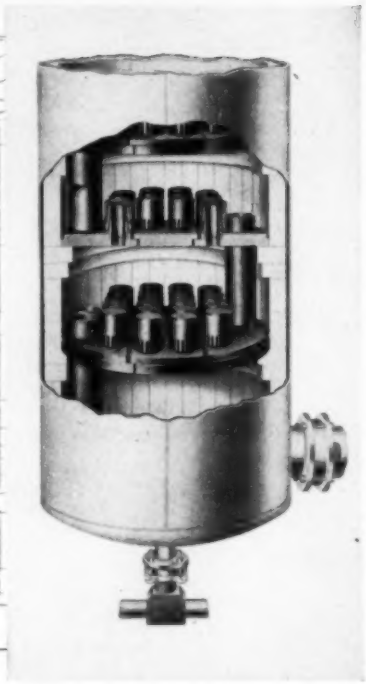

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# OPINION AND COMMENT

## COMMUNICATIONS—AN AID TO COOPERATION

IN spite of all that has been written on the subject, there is still a definite need for emphasis on the advantages of cooperative effort in bringing an engineering project to full satisfactory completion. This is particularly directed at our younger engineers. However, there are times when the rest of us also need reminding.

Every young engineer, as he comes out of school, is fired with ambition, thoroughly convinced that he knows all the answers from a technical point of view. He will drive ahead on his assigned job without looking to right or left, and eventually be hauled up short by his supervisor because he has failed to consider some practical angle that a little consultation with his associates would have made self-evident. If he continues to work in this fashion, he will find sooner or later that his usefulness is limited and his progress slower than his more cooperative fellow employees.

Every list of requirements for success in the engineering profession contains, fairly near the top, the ability to work with others. This, of course, primarily means ability to get along with your supervisor, your close associates, and with those whom you may be called upon to supervise, but in the long run this must be broadened to include the ability to work with people in other functions of your business, who ordinarily have quite different points of view from that of the average engineer.

As an engineer matures, he will find increasing advantage in exchanging points of view with others in different kinds of work. His tendency, at first, is to be scornful, particularly of the practical man, but if he is alert he will soon realize that he gains by every such exchange of ideas. A willingness to listen to the other fellow and to be tolerant of his point of view is an attitude which is well worth cultivating for it will pay dividends many times over. One fatal mistake that many engineers make is to assume that discussing his problems with someone else will take away from the credit that he expects to receive from the successful completion of his assignment. Nothing could be further from the truth. A smooth working team that produces results receives ample credit for everyone, with plenty to spare.

The magnitude of most industrial projects today is such that a wide diversification of technical skills is required for successful completion. The smoothness and effectiveness with which these skills are blended is in large part responsible for the degree of success enjoyed. The exact nature of the communication system which is used to foster this kind of cooperation is largely dependent on the size, variegation, and geographical arrangement of the organization. In general, the less formal the procedures that are set up, the more likely are the results to be successful. Where there are natural barriers to be broken down, however, it is often necessary to set up semiformal procedures, such as meetings at stated intervals, or reviews of projects at certain stages of completion.

This problem is particularly difficult in large research and development organizations which have outgrown the situation where everyone knew what everyone else was doing. For the most part, the methods used in manufacturing operations are not too applicable to the administration of research and development work, where operations are directed toward new things rather than the efficient repetition of well-known things. Personal experience in a medium-sized research and development department would indicate that satisfactory results can be obtained if those in charge of a development during its initial stages will discuss problems as they occur with the engineers further down the line rather than waiting to pass on a completed package. The reverse procedure, when the project nears completion, will also bear fruit. Occasional more formal presentations at logical points in the development are also advantageous. They serve a double purpose; first, to educate all interested personnel and, second, to increase the prestige and experience of the individual who makes the presentation.

The specific problem of communications in research and development work is of such importance that it is presently the subject of a research study at one of our well-known institutions of learning. Many managers of research and development departments across the country who have wrestled with this problem will be glad to see the results when they are published.

The engineer who wishes to reach a responsible position in his company must learn to search for and make use of the points of view of all those concerned with the problem at hand. It is only by such cooperative effort throughout all levels of engineering organization that management can obtain the smooth and effective operation which is characteristic of successful companies.

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# GALVANIC CORROSION

E. C. REICHARD

American Smelting and Refining Company, Perth Amboy, New Jersey

*We begin this month a series of papers on Corrosion, a topic of prime importance to the chemical process industries. . . . In this issue E. C. Reichard discusses galvanic corrosion, and points up the ways to prevent costly failures within chemical plants. . . . This will be followed in a month with another article on Corrosion by E. C. Tice, and the series will be concluded in the August issue with a story on corrosion testing in which E. G. Holmberg will discuss the importance of research leading to new alloys—which ultimately means conservation of metals.*

CHEMICAL plants are conglomerates of various metals and nonmetallic containers, interconnected by elaborate systems of piping. In these are handled fluids of many kinds, concentrations, and all degrees of aeration and temperature—just the ideal requirements for corrosion, especially galvanic corrosion.

**Galvanic Cell.** To have a galvanic cell, it is essential to have an electrolyte in contact with two different metals or electrodes that are metallically connected to each other. The galvanic cell, as applied in the common flashlight battery, is so familiar that the principles upon which it is constructed are often ignored. The galvanic current produced from connecting carbon with zinc provides a 1½-v. source of electricity, the current which is used daily in lighting lamps and actuating relays (*see* Fig. 1).

Chemical engineers build galvanic cells every day with various pieces of equipment. Every cell that is made, accidentally or intentionally, produces its own measurable current. Whenever this current leaves a metal and enters an electrolyte, it takes some particles of metal with it. This is called the anodic reaction of the galvanic cell and is described often as the place where a metal ionizes and leaves an excess of electrons.

Each cell has another portion at which a cathodic reaction takes place. The metal is protected; no corrosion normally occurs at the site of the cathodic reaction. The cathode of a galvanic cell is similar to the cathode upon which other metals are deposited in the electrolytic refining industry and in the plating shop. At the anode of a galvanic cell, metal is destroyed, whereas at the cathode metal is protected. Characteristics of the anodic phase of the corrosion reaction warrant closer examination.

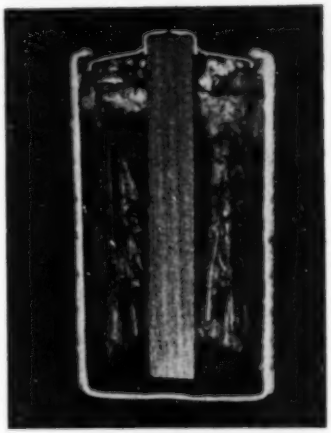


**Fig. 1. A useful galvanic cell. The common 1½ v. dry cell consisting of zinc case and carbon rod.**

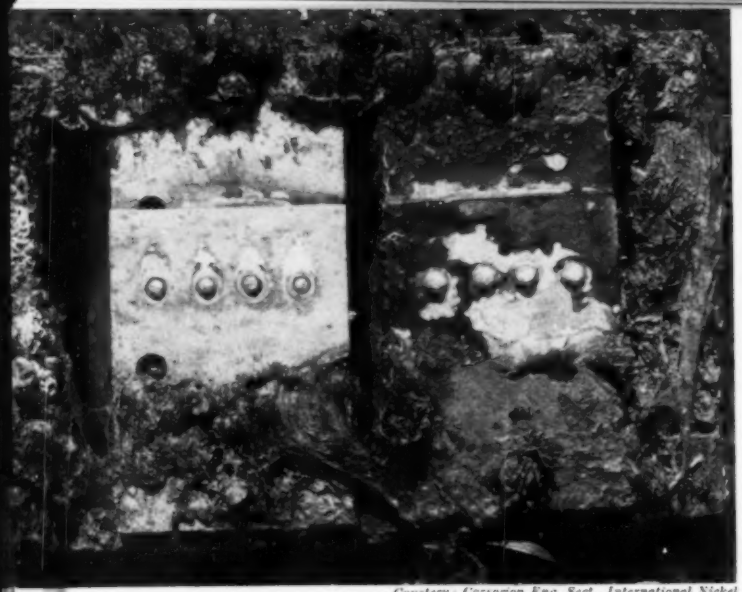
**Keep Anode Areas Extensive.** The current density at the anode determines the corrosion rate. Reduction of the anode current density produces less severe galvanic corrosion. When it is necessary to have two metals, the aim should be to have large areas of the anodic metal and smaller areas of the cathodic material. Thus it is better to have steel sheets fastened with copper rivets rather than the reverse.

In 1915, steel rivets were accidentally used in fastening monel sheathing on the now famous yacht the Sea Call. This vessel was scrapped a few weeks after launching because of the failure of the steel rivets. These rivets had suffered from galvanic corrosion. The small and intense anode areas of the rivets had been surrounded by extensive cathode areas of monel. Figure *2* shows the galvanic corrosion of steel rivets when used in copper plate and the lack of intense corrosion when copper rivets are used for steel plate.

**Paint Cathode Areas.** In some cases, the undesirable effects of galvanic action can be prevented by the application of suitable paint coatings to metal surfaces. It should be pointed out, though, that painting of the anodic metal surfaces alone can be a dangerous practice. This simply reduces the anode area and allows intense galvanic action to occur at any breaks or discontinuities in the paint film. If painting is to be used, the cathodic areas should be painted first and the paint films maintained on these areas. The cathodic areas are not damaged by galvanic action and, therefore, can be safely used in controlling the action. If the paint film fails on the cathode, then the former cell was only partially renewed, but its action was not intensified.

Recently a major automobile manufacturer painted steel radiator headers and soldered them to the usual copper radiator core. He was trying to conserve critical copper, but instead ended up with a costly galvanic corrosion problem. Leaks developed in these radiator headers. The extensive noble metal areas of the copper core, metallically connected to the limited anode areas resulting from breaks in the paint

*E. C. Reichard has been employed since World War II as a research metallurgist, specializing in corrosion problems in the central research laboratory of the American Smelting & Refining Co., Perth Amboy, N. J. Prior to this association he was employed by the Phelps Dodge Copper Products Corp., Bayway (New Jersey) Mills, and Picatinny and Frankford Arsenals before going on active duty in the Naval Reserve. Mr. Reichard, a native of New York City, is a graduate of the school of engineering, Columbia University, New York.*



*Courtesy: Corrosion Eng. Sect., International Nickel*

**Fig. 2. Area effect shown by rivet and plate assemblies.**

Copper plates, steel rivets. Steel plates, copper rivets.

coating, caused an active galvanic cell. A galvanic cell of lower intensity would have been obtained if no paint had been used on the steel headers.

The manufacturers undertook to replace the defective radiators. This replacement job was estimated by the manufacturer to cost $20 million (*2*) to recall and replace the radiators of more than 200,000 automobiles—a costly error to rank with the Sea Call story.

**Specify Anodic Coatings.** Sprayed metallic, hot-dipped, and electroplated coatings can be used also to prevent undesirable galvanic action. Here again, it is safer to work with the cathodic material and coat it with a more anodic metal. Galvanizing and cadmium plating are steps in the right direction. The failure of decorative chromium plating is an example of the reverse situation. Here the pores in the chromium plate allow small areas of iron, the relatively anodic metal, to be exposed and unsightly staining results.

**Break Metallic Paths.** Galvanic corrosion can be halted if the metallic path between the anodic and the cathodic reactions can be broken. Gas companies are now isolating their steel distribution lines from contact with the lead and copper water services (*6*). These lines of steel and copper are generally tied together in a galvanic cell through the hot water heater. Gas companies are now installing insulating coupling at their meters to break this galvanic cell.

**Add Inhibitors.** In recirculating systems inhibitors can be used to control the destructive effects of galvanic cells.

Let us return to the automobile with its large area of copper found in the radiator. This is connected by rubber hose to less extensive areas of cast iron or aluminum in the cylinder assembly. This rubber hose does not isolate the copper from the block if the radiator and engine are attached to the car frame by simple bolting. Again this is a common electrolyte, in contact with two different metals, that are tied to each other through the car frame. The galvanic cell could be broken by insulating the radiator from the car frame, but such is not practical. In this particular problem, inhibitors provide an ideal solution because of the recirculating nature of the coolant.

Financial reports of a fire-extinguisher manufacturer show that $1,225,000 (*1*) was charged directly to corrosion losses. These corrosion losses occurred because of the use of fire extinguisher fluids that corroded the valve components of the extinguishers and caused them to jam. So 500,000 fire extinguishers were recalled and replaced with new ones with a fluid containing an inhibitor.

Many organic compounds act as inhibitors, if they are to be added as a part of regular process operations. They are added as early in the process as possible to obtain the benefit of their inhibitive effect.

**Galvanic Series.** Thus far the effect of areas, altering a metal surface with paints or other coatings, and breaking the metallic circuit has been considered. The question of which metal is the anode may present itself. On an operating problem, the metal that is corroding is definitely an anode; on a design problem, judgment must be exercised. The galvanic series is usually arranged as shown in Table 1.

**Pitfalls of Galvanic Series.** It is wise to beware of conclusions drawn from data in Table 1. Results are based on ideal laboratory conditions not attainable in normal plant operations. The stainless alloys, under certain conditions of service, act like the noble metals. Yet slight changes in those conditions cause them to act like ordinary iron so far as galvanic corrosion is concerned and they are then called active rather than passive.

Unusual instances have been reported (*7*) in which chromium-nickel-molybdenum alloys have three positions relative to a standard of comparison. It is not always realized that similar alterations are possible with other metals.

Zinc is often used in practice to protect aluminum equipment. Accordingly many tables now place zinc over aluminum instead of below. Zinc in galvanized hot-water tanks will be an anode and protect the steel tank provided temperatures do not go too high. Yet a demand for very hot water induces a reversal of the usual galvanic relationship and the zinc becomes the cathode surrounding smaller exposed areas of the underlying steel (*3*). Here again the unfavorable area relationship occurs. An extensive galvanized surface acting as a cathode and a few spots of exposed iron acting as anodes result in premature perforation of the hot-water tank.

A word might be given at this point concerning lead. It is rarely a factor in galvanic corrosion because it becomes covered with electrically inert sulfate films. In any case, lead shifts to a nobler or safer potential so far as it is concerned.

From these comments it is evident that the galvanic series must be used with caution because most metals can shift their position depending on environment.

**Cathodic Protection.** A metal can intentionally be used that acts as an anode to protect critical components and "buck out" other galvanic and concentration cells. This is done, to a certain extent, when galvanized or cadmium-plated steel and pure aluminum clading over stronger cathodic aluminum alloys (Alclad products), are specified.

A certain plant in New Jersey recently wanted to control the corrosion on condenser heads. No trouble was being encountered with the copper base alloy tubes, so it was reasoned that painting of the iron surfaces would reduce their corrosion. Shortly after the paint had been applied, the operators were surprised with a pitting failure of the heads. Previously the attack had been distributed over the entire conden-

ser head surfaces; it was relatively uniform and considerable service was nevertheless obtained. By painting the anode, the attack was localized and intensified to the points of the failure in the paint coating.

Painting the tubes, which were the cathode, adding inhibitors, or breaking the metallic circuit were not practical procedures in this problem. Painting the anode was tried, but this step only accentuated the trouble. A satisfactory solution finally was found by fastening pieces of magnesium to the insides of the inspection plates. These pieces of magnesium corrode rapidly, lasting just about a year and are replaced during the annual inspection of the condenser. They do the valuable job of diverting the corrosion from the costly condenser heads. Typical sacrificial anode shapes are shown in Figure 3.

Pieces of zinc and magnesium are being used now to protect many large vessels and are also being attached to piping systems to protect them from external corrosion. Corrosion prevention is important on the large transcontinental pipe lines which bring oil and gas from Texas to the East. For example, a 30-in. pipe line has only a 5/16-in. wall thickness. The overdesign of this wall thickness by only 1/16 in. would add 92,000 tons of steel and increase the original cost or investment to $20 million (using 1950 prices) (*4*). Yet if no other protection is added, increasing the wall thickness only delays temporarily the appearance of leaks and corrosion damage. Current design practices are such that loss of 1/16 in. of the parent metal of a 5/16 pipe line would result in its operating at 100 per cent of the yield strength of the metal. Corrosion is controlled on these lines by coatings to reduce the cathode areas and then by placing sacrificial pieces of magnesium or zinc in metallic contact with the line to protect or "buck out" the anode areas. The corrosion is diverted thus to noncritical and expendable anodes. If corrosion can be controlled on pipe lines extending more than a thousand miles in length, there seems little excuse for the repeated failures of piping that occur in your plant and mine.

Many plants are now installing sacrificial anodes in a routine manner when repairing leaks in their utility and service lines. After all, a $100 a day consulting corrosion engineer accoutered with elaborate instruments is not required to determine that pipes are corroding after a leak has developed. A leak is a telling argument that protection is needed in a given area, even if and after it is repaired. Sacrificial anodes installed during the repair provide the least expensive type of protection obtainable. Protection of this type is called

TABLE 1.—METALS IN GALVANIC SERIES

Corroded End (anodic or least noble)

Magnesium
Aluminum
Zinc
Cadmium
Iron or Steel
Chromium-iron (active)
Stainless Steels (active)
Soft Solders
Tin
Lead
Nickel
Brasses
Bronzes
Nickel-Copper alloys
Copper
Chromium-iron (passive)
Stainless Steels (passive)
Silver
Carbon
Gold
Platinum

Protected End (cathodic or most noble)

"hot-spot" protection. If enough sacrificial metal is installed, galvanic corrosion will be completely arrested. If sufficient current is not obtained from the sacrificial metal, full protection will not be obtained, and the rate of corrosion will be reduced.

## Summary

A number of everyday examples have been touched upon. These are typical and can point the way to preventing costly failures within chemical plants. To minimize accelerated damage from galvanic corrosion the following procedure is recommended assuming that different metals must be used.

1. Keep the anode area more extensive than that of the cathode. Copper rivets in steel plate or remember the "Sea Call."

2. In applying coatings and barriers of other metals, work with the cathodes first so that damaging effects will not be accentuated (condenser heat problem).

3. Break metallic connections or paths wherever possible.

4. Specify anodic coatings—galvanized steel and Alclad.

5. Add inhibitors to recirculating systems (automobile radiator example).

6. Beware of generalizations from the galvanic series which is based on ideal laboratory conditions.

7. In a galvanic couple that can't be avoided, don't use anodic metals for the critical structural material like the common dry cell or the "inside out" battery.

8. Use sacrificial anodes of zinc and magnesium to have the galvanic cell work to your advantage in giving cathodic protection to critical equipment even if only hot-spot protection is obtained.

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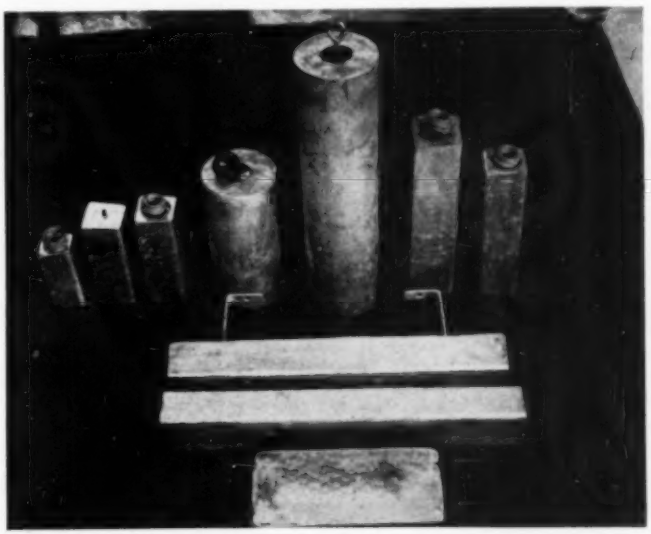

**Fig. 3. Typical forms of sacrificial magnesium anodes.**
**Top, left to right: 3-lb. service anode; 12-lb. flush top; 12 lb.; 50 lb.; 100 lb.; 32 lb. and 17 lb. Bottom: back to front: 40-lb. tank anode; 36 lb.; and 16-lb. hull anode.**

*The first of four articles on ammonia synthesis begins in this issue with H. A. Tuttle's development of the economics of using hydrocarbon gases as a source of material for synthesis gas. . . . The second article will be by Yeandle and Klein who will cover commercial processes for purification together with descriptions of typical equipment installations. . . . L. B. Hein in the third article will follow with an engineering description of ammonia synthesis processes which operate at 350 atmospheres . . . and the final paper by Thompson, Guillaumeron and Updegraff will discuss the present-day Claude process. . . .*

# PREPARATION OF CRUDE SYNTHESIS GAS FROM HYDROCARBONS

H. A. TUTTLE

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**This paper develops the economic reason for using hydrocarbon gases as a source of material for ammonia synthesis raw gas. Thermal cracking is discussed briefly; reaction rates are given, and limitations and disadvantages of the process are pointed out. The catalytic process is discussed in more detail with stress on the function and design of the main items of equipment. Correlation of these and other auxiliary equipment is explained and their relationship shown by a flow diagram.**

THE increasing demand for fixed nitrogen throughout the world has stimulated the production of hydrogen for the synthesis of ammonia beyond all expectations. The most abundant sources of hydrogen for this purpose are water and the hydrocarbons. There is, to be sure, an adequate supply of water available everywhere and a quite plentiful supply of hydrocarbons in most parts of the world. However, it requires extensive processing of these raw materials to obtain free hydrogen. In this paper there will be a discussion on (1) the economics of using the hydrocarbon gases and, (2) methods of reforming these gases into a raw gas suitable for conversion into ammonia.

**Economic Consideration.** Due to its abundance, water is by far the greatest competitor with the hydrocarbons as a source of hydrogen. Hydrogen may be obtained from water, either by electrolysis or by the reaction of water-vapor with a solid, a liquid or a gaseous carbonaceous material. Although the electrolysis of water or of aqueous salt solutions provides the purest hydrogen, it requires a cheap supply of electric energy. This usually precludes the consideration of electrolysis—except where other forms of energy are wholly lacking—or where the hydrogen is the by-product of an electrolytic process used to obtain other products such as chlorine and caustic.

Use of solid carbonaceous fuel and water vapor has been in the past the more accepted method of obtaining hydrogen (witness the use of gas producers for gasification of coke or coal with steam). More recently use of hydrocarbon gases has been playing a rapidly expanding role in supplying hydrogen to the producers of ammonia. The reason for this is purely economic as may be shown by comparing the relative costs of the raw materials.

To produce the hydrogen necessary for one short ton of ammonia requires approximately 35,000,000 B.t.u.'s. Translating this into the cost of raw material or energy, it is equivalent to 10,450 kw.-hr./ton of ammonia using electrolytic process or 1.4 tons of average coke/ton of ammonia or 35,000 cu.ft. of natural gas/ton of ammonia. With natural gas selling at 20 cents/M. cu.ft., coke would have to cost $5.00/ton and electricity 7/10 mils/kw.-hr. to be competitive.

This favorable economic position for

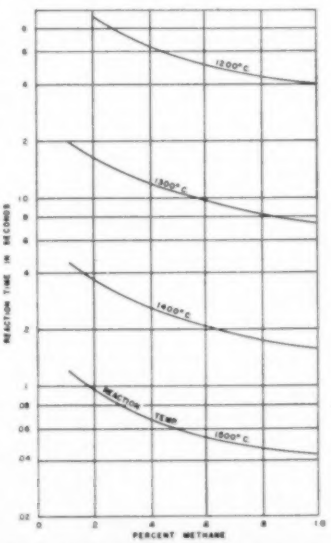


Fig. 1. Reaction rate curves for methane steam thermal cracking.

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natural gas has stimulated the development of processes for the conversion of the hydrocarbon gases (chiefly natural gas) into hydrogen.

While the hydrocarbon gases may be processed with oxygen alone to produce hydrogen, the reaction of hydrocarbon gases with steam has the added advantage of obtaining hydrogen both from the reacted gas and the decomposed steam.

Two methods are available for carrying out this latter reaction:

1. The thermal cracking of hydrocarbons in the presence of steam.
2. The catalytic reforming of hydrocarbon gas with steam.

**Thermal Cracking of Hydrocarbons.** In the thermal cracking of hydrocarbons the reaction rates for the higher hydrocarbons are so rapid that the rate for methane predominates and is the determining factor in the design of process equipment.

Figure 1 gives the reaction time for the reaction $CH_4 + H_2O \rightarrow CO + 3H_2$ when using the thermal cracking process. It is apparent from these curves that temperatures in excess of 1300° C. must be employed in order to maintain the equipment at a reasonable size. This involves the use of high-temperature refractories and the regenerative cyclic-type process. Many processes of this type have been developed which have proved costly to build and expensive to operate. To overcome this disadvantage the catalytic process has become the almost universally accepted method of reforming hydrocarbon gas to obtain hydrogen.

**Catalytic Reforming of Hydrocarbon Gas.** The basic equations for the catalytic reforming of methane with steam are

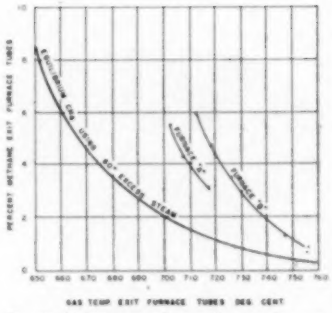



Fig. 2. Performance of gas-reforming furnaces using natural gas.

$$CH_4 + H_2O \rightarrow CO + 3H_2 - 49269 \text{ cal./g. mole} \quad (1)$$

$$CO + H_2O \rightarrow CO_2 + H_2 + 9839 \text{ cal./g. mole} \quad (2)$$

Similar equations may be set up for the higher hydrocarbon gases and indeed they must when considering such raw materials as natural gas, refinery gas or coke-oven gas. As with thermal cracking the reaction rates for the higher hydrocarbons when using the catalytic process are so much faster than for methane that as regards equilibrium conditions we need to consider methane only.

On Figure 2 the equilibrium methane concentration is plotted for natural gas reforming as a function of temperature when operating with a steam to gas mole ratio of 0.5 in the product gas. To indicate the approach to equilibrium that may be expected with catalytic reforming, curves have been included taken from two reforming furnaces operating within this range of temperature and with the same steam to gas ratios. It is, of course, understood that Equations (1) and (2) must satisfy equilibrium conditions at the considered temperature.

Use of the catalytic process reduces the reaction temperature to a degree where high temperature alloy metals may be used. Also the reaction rates have been so increased that the time element no longer controls but heat transfer and pressure drop predicate the design features of the equipment.

The over-all reaction is highly endothermic and requires considerable quantities of heat to raise the reactants to the operating temperature and to maintain the reactions. Some economy may, of course, be realized by proper heat transfer between the product gases and the reactants. Also heat-recovery equipment, such as waste heat boilers and combustion air preheaters, may be used to reduce further sensible heat losses.

Figure 3 shows a flow diagram for an accepted gas-reforming installation wherein the nitrogen required for the process is obtained by burning a controlled amount of air in what has been called here a secondary reformer. Flue gas of suitable purity may be substituted for the air if desired.

**Primary Reformer.** What is termed the primary reformer on this flow diagram is the central figure of the process and deserves special attention. A number of designs have been developed for this piece of equipment, all of which have certain fundamental functional features in common. It is the detailed arrangement of specific elements in the construction of the reformer which differentiate between the designs.

The almost universally accepted version of the primary reformer consists of a furnace, in which are placed vertical high-temperature alloy steel tubes containing the catalyst, together with the burners to provide the heat required to maintain the reaction.

The tubes are generally of chrome nickel stainless steel type 310 having a diameter of from 6 to 8 in. and a length

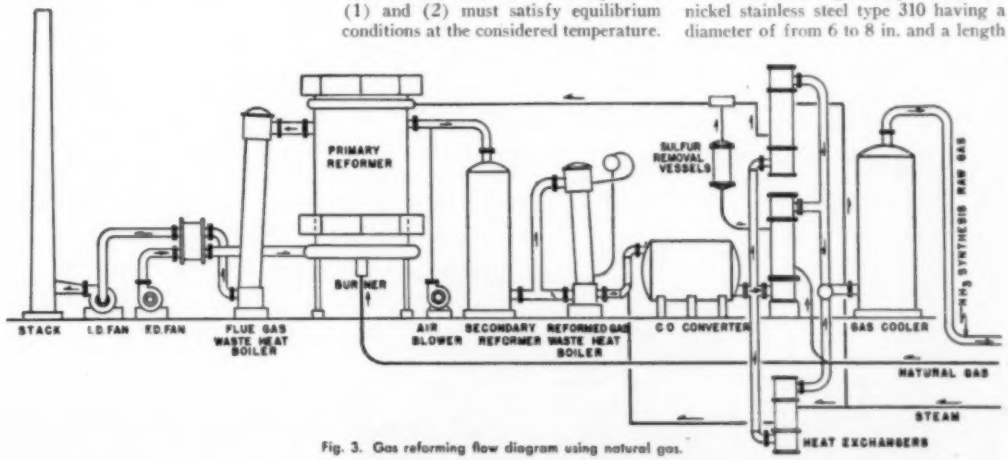



Fig. 3. Gas reforming flow diagram using natural gas.

of 20 ft. or more. The catalyst, a promoted nickel oxide, is supported on a grate within the tube to a maximum depth permitted by the tube length. Tubes are operated in parallel, the flow to each being equalized by sized-orifice restriction plates or other suitable regulatory devices. Mounting of the tubes within the furnace must be such that it allows for thermal expansion and provides uniform heating of the tube circumferentially as well as longitudinally. A 20-ft. long tube having a wall temperature of 900° C., which is not unusual, would expand longitudinally at least 4 in. while the uneven heating will cause it to distort and create unnecessary stresses in the metal.

Figure 4 shows a reforming furnace in which the expansion is taken care of by suspending the tubes from the top and allowing them to swing freely. No bottom connections to restrict the lateral movement is involved. Gas flow is maintained through the catalyst by admitting the reactants at the top of the tube and releasing the product gas from below the grate through a central pipe which passes up through the catalyst and out the top.

Even heating of the tubes is assured by having the rows arranged in the form of a cross with burners placed evenly on either side of the rows in the floor of the furnace. The waste flue gases are withdrawn from the top of the furnace and pass on to a waste heat boiler and combustion air preheater.

The product gases leaving a primary reformer operating at 700-720° C. catalyst temperature would have a composition about as follows:

$CO_2$—11.5 mole %
$O_2$— 0.1 mole %
CO—10.5 mole %
$H_2$—77.0 mole %
$CH_4$— 0.3 mole %
$N_2$— 0.6 mole %

It is apparent that this gas must undergo further processing before it is suitable for ammonia synthesis raw gas. The CO content must be reduced to a minimum and nitrogen added to provide the proper ratio of hydrogen to nitrogen. This may be accomplished in a number of ways.

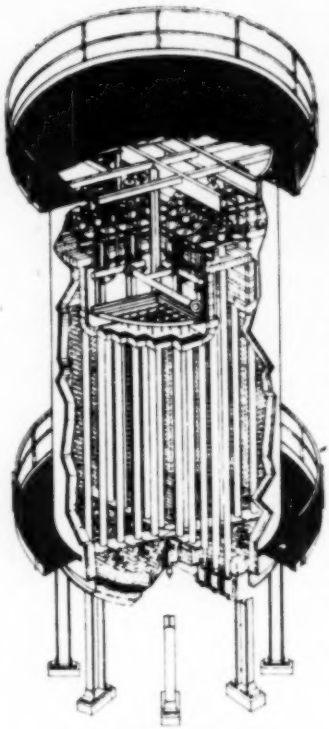


Fig. 4. Primary reformer heater.

The primary reformed gas may be reduced to a temperature favoring the reaction $CO + H_2O \rightarrow CO_2 + H_2$ and passed over a shift catalyst accompanied by additional steam whereby a large portion of the CO would be converted to $CO_2$ and additional $H_2$ obtained. This gas may then be mixed with nitrogen derived from an inert gas generator or liquid air nitrogen producer.

**Secondary Reformer.** The flow sheet shows a secondary reformer interposed between the primary reformer and a waste heat boiler. This piece of equipment serves the dual purpose of relieving the load on the primary reformer and provides a means of adding nitrogen to the gas stream.

Air is admitted to the gas stream entering the secondary reformer and burns with the primary reformed gas. The oxygen is thus consumed leaving the nitrogen to mix with the process gases and raise its temperature before passing over a catalyst, similar to that in the primary reformer.

A temperature rise of as much as 300° C. may be expected in this gas from the combustion of the nitrogen air. This permits us to reduce the temperature of the primary reformer either by increasing the reformer throughput or by reducing the fuel consumption or both. The methane content of the primary reformed gas will increase and may be permitted to rise to 6 or 7%. The secondary reformer will reduce this to the acceptable figure of 0.2 to 0.3% methane. A characteristic secondary reformed gas would be as follows:

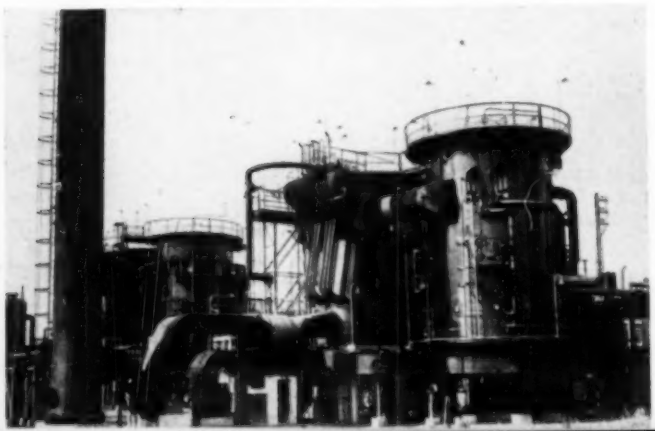
$CO_2$—10.5 mole %
$O_2$— 0.1 mole %
CO—11.5 mole %
$H_2$—57.0 mole %
$CH_4$— 0.3 mole %
$N_2$—22.6 mole %

**CO Conversion.** The final step in the processing of the hydrocarbon gases is to reduce the carbon monoxide to an acceptable and economic value. If one examines the equilibrium constants for the reaction $CO + H_2O \rightleftarrows CO_2 + H_2$, it is found that lowered temperatures and excess steam favor the formulation of $CO_2$ and $H_2$.

Unfortunately the lower temperatures necessary to obtain acceptable CO concentrations depress the reaction rates to such an extent that a catalytic process is mandatory. Here again lowering the temperature decreases the activity of the catalyst so that a compromise must be reached between operating temperature and degree of conversion desired. Temperatures of from 350 to 500° C. are usual practice and a CO concentration of from 2-4% is an accepted value.

Inasmuch as the CO conversion reaction is slightly exothermic the temperature of the gases from secondary reformer must be reduced to about 50° C. below the reaction temperature desired in the CO converter. This may be done in a number of ways; by water quenching, admission of excess steam or by a

Fig. 5. Gas-reforming plant for 200 tons/day $NH_3$ plant, showing primary reformer, flue gas waste-heat boiler, air preheater, forced and induced draft fans and stack.



waste heat boiler, or by combinations of these means.

As to the CO converter itself, it can be of the simplest design requiring only a carbon steel vessel with a grate to support the catalyst. The dimensions are determined by the allowable pressure drop and the required space velocity.

The final converted or raw ammonia synthesis gas from such a process would have an average composition of the following

$CO_2$—17.0 mole %
$O_2$— 0.1 mole %
CO— 3.0 mole %
$H_2$—60.0 mole %
$CH_4$— 0.3 mole %
$N_2$—20.0 mole %

As noted from the flow sheet heat is recovered from the final converted gas by means of heat exchange with the incoming reactants, while final cooling of the product gas is accomplished by water washing. A desulfurization unit is indicated. This is a precautionary measure since sulfur compounds act as a major deterrent to the efficient performance of the reformer catalyst. Since pressure drop is a deciding factor in the design, it can be stated that a pressure of from 50 to 75 lb. gage is adequate to overcome the resistance of the system and deliver the gas to the gas holder.

## Discussion

**J. J. Dorsey, Jr.** (Monroe, La.): To what degree do you limit the tube wall temperatures in your reformer?

**H. A. Tuttle:** I try to keep them below 850° C.—that is the shell temperature of the tube—850 to 900° C. Some of them have been operated as high as 1100° C. but it is not very good for the tubes.

**J. J. Dorsey:** Do you advocate use of boiler flue gas as a nitrogen source for ammonia synthesis? Do you base your opinion on the economics of the method?

**H. A. Tuttle:** Usually the hydrogen is more valuable as a source of raw material for ammonia than it is as a fuel for making nitrogen. If you are interested only in the amount of ammonia you can make and you have a lot more hydrogen available than you need, then you burn the hydrogen and make nitrogen by burning the oxygen out of the air.

**C. D. Wrigley** (Philadelphia, Pa.): If a stream of fairly pure hydrogen were available, which was the by-product of some other process, what, in your opinion, would be the best way to introduce nitrogen to make an ammonia synthesis gas?

**H. A. Tuttle:** By air liquefication.

**J. I. Hughes** (Charleston 4, W. Va.): You mentioned the use of methane and steam in making these gases by reforming. Can you use other hydrocarbons such as LPG, a butane-propane-methane?

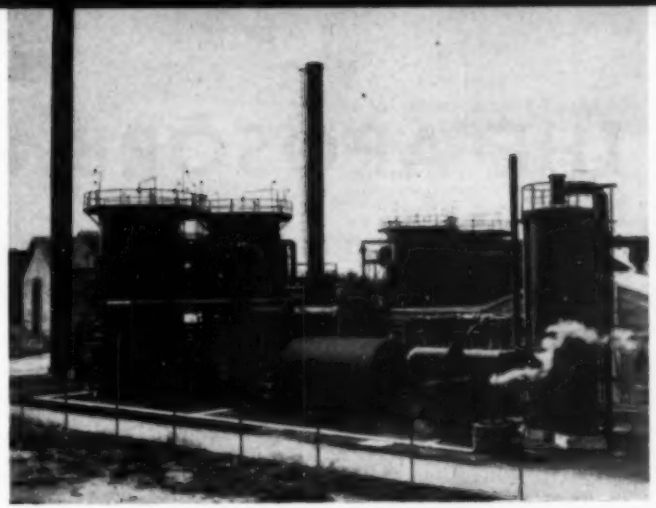


Fig. 6. Gas-reforming plant for 200 tons/day $NH_3$ plant, showing primary reformer, secondary reformer, reformed gas waste-heat boiler, carbon monoxide converter, heat exchangers and gas scrubber.

**H. A. Tuttle:** Yes. We use natural gas in all its combinations. Also we use refinery gas, coke-oven gas. We use methane as an indication of the process because all these higher hydrocarbons react so much more rapidly that the thermodynamics and kinetics of the reaction can be based entirely on methane.

**J. I. Hughes:** Does the hydrogen and CO ratio change much when you use a higher hydrocarbon?

**H. A. Tuttle:** The product gas must and does satisfy the water-gas reaction.

**J. I. Hughes:** About using so much heat indirectly in this process outside of the tubes—Is there any possibility of introducing at least part of the endothermic heat required by introducing air into your primary reformer, in some way?

**H. A. Tuttle:** Yes, there are patents covering that. In this process it is being done in the secondary reformer.

**J. I. Hughes:** Can you do it all in one?

**H. A. Tuttle:** You can. There are processes doing that. Also there are processes whereby the flue gases are admitted with the steam and reaction gas.

**W. E. Lobo** (M. W. Kellogg Co., New York): You mentioned the importance of even heating of the tubes. Have you ever measured the conversion in the various tubes of the furnace, or the tube metal temperatures of all tubes and is the flow uniform in all the tubes?

**H. A. Tuttle:** Yes we have. The flow is quite uniform except when somebody mismanages the operation and turns the steam off, then carbon is deposited on the catalyst and decomposes the catalyst. We then get variations in flow through the various tubes. In order to get uniform flow through the tubes and just in case such an occasion arises, we put an orifice plate into each tube and that levels off the flow. I have here data taken in the Mexican plant which the chairman mentioned. When we started up, the catalyst temperatures in the tube banks were 655-656-655-651. That is a pretty even temperature and the average temperatures of gases coming out of the tubes were 545-535-524-535. We had even distribution. Of course you can undo this even distribution by not having your burners adjusted properly. You can overheat one bank of tubes and underheat another. The catalyst breakdown is one problem with the particular type of catalyst that the Chemical Construction Co. uses. If you get a carbon deposition on your catalyst it certainly doesn't improve it any. However, it still remains a good catalyst but increases the pressure drop and causes uneven flows. It is more like a ceramic than anything else. Some of the precipitated catalysts on carriers are not subject to this breakdown but they are not quite so active.

**A. G. Eickmeyer** (J. F. Pritchard & Co., Kansas City, Mo.): Are there any steam-methane reformers operating at substantial exit pressures, that is, above atmospheric?

**H. A. Tuttle:** Not commercial ones.

**A. G. Eickmeyer:** Experimental?

**H. A. Tuttle:** Experimental—yes about 30 lb. and that is not much of a pressure.

*(Presented at A.I.Ch.E. Kansas City (Mo.) Meeting.)*

# THE AROSORB PROCESS

JAMES I. HARPER, JOHN LEE OLSEN, and FRANK R. SHUMAN, JR.

Sun Oil Company, Marcus Hook, Pennsylvania

**This short historical review of the Arosorb process is accompanied by a simplified flow sheet to give an over-all concept of the process. Process variables and other chemical engineering factors entering into the design of an Arosorb unit are discussed. Underlying principles of commercial hydrocarbon separation by silica gel are presented, and the factors involved in setting up a silica-gel case cycle for a typical feed stock are covered. The influence of percolation rate, pressure drop, height, diameter and number of cases is considered in relation to the shape of silica-gel beds. A description is included of the silica gel used and the expected life. Instrumentation, where peculiar to the Arosorb process, is described.**

A PROCESS, known as the Arosorb, is a commercial application of the selective adsorptive power of silica gel to separate various types of organic compounds, particularly aromatics from hydrocarbon streams. The separation of hydrocarbons according to chemical type, by use of silica-gel adsorption, has passed through the laboratory and pilot-plant stages and is now entering commercialization. Silica-gel adsorption was successfully used in the laboratory for hydrocarbon analysis in API Project No. 6 under the direction of Rossini and Mair (*5, 4*). The method was generally accepted as a laboratory tool, but resulted in no commercial application. Lipkin (*3*) conceived a modification of the procedure which opened up possibilities for a large-scale process. Development over the years has brought the operation to the present commercial stage and the name Arosorb has been coined to designate the process. It is covered by patents and patent applications of Sun Oil Co.

The process is flexible for handling hydrocarbons of almost any boiling range, and even nonhydrocarbon streams. A typical example is given of separating benzene and toluene from the paraffins and naphthenes in a suitable petroleum fraction. The simplified flow sheet (Fig. 1) shows how the process operates:

A nominal thousand barrels per day of charge stock after passing through driers enters one of several silica-gel beds for a period of about 30 min. During this portion of the cycle the aromatics in the charge stock are adsorbed by the silica gel. The saturate portion of the charge is pushed out of the bed by a so-called "push liquid," which in this instance could be butane or pentane. This step requires another 10 min. Then a stream of xylenes is added for a period of about 40 min. to desorb or remove the aromatics from the silica gel, regenerating the latter for the next cycle.

For about two thirds of the cycle the effluent contains the saturates from the charge stock. During the other third the effluent contains the charge aromatics. At suitable times during the cycle, valves are switched to segregate the saturate effluent from the aromatic. Both streams contain push liquid and desorbent and the streams are sent to distillation columns to recover these liquids which are recycled. The saturate effluent passes first to T-1 for recovery of push as overhead, with the bottoms passing to T-2 where the saturate product is recovered as an overhead stream and the desorbent as a bottoms. The aromatic effluent goes to T-3 for recovery of push liquid and then to T-4 where a stream of benzene and toluene is taken overhead and the desorbent xylenes out the bottom. T-5 is merely a benzene-toluene splitter.

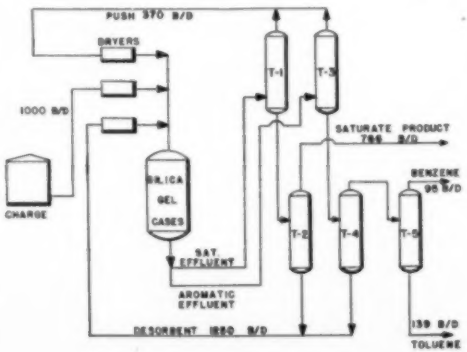



Fig. 1. Simplified flow sheet for Arosorb process.

## Underlying Principles

Silica gel adsorbs aromatics preferentially to olefins or saturates. The adsorption equilibrium diagram for the system benzene-*n*-heptane (Fig. 2) was determined by contacting silica gel with various benzene-*n*-heptane mixtures and noting the change in liquid composition at equilibrium. The benzene adsorbed, as determined from the change in liquid concentration, is called the apparent volume adsorbed, and does not account for all the liquid necessary to fill the total measured pore volume (*1*). The adsorbed phase composition (Fig. 2) is defined as the concentration of benzene in the silica-gel pores and is calculated by adding to the apparent volume of benzene adsorbed the material filling the balance of the pore space. By this method of calculation the latter liquid has the same composition as the interstitial liquid. Diagrams for olefin-saturate mixtures obtained in a similar manner show the olefins are less strongly adsorbed than aromatics. Equilibrium diagrams for systems composed of the same chemical type, such as saturate-saturate, can be calculated from published data (*2*) and are found to be lines having approximately 45° slopes.

Assume that a stock consisting of 27% benzene and 73% saturate is fed to a dry silica-gel column and that the quantity of feed is less than the adsorptive capacity of the gel. This will result in the schematic composition diagram (Fig. 3). The right-hand portion of the diagram gives the per cent benzene in the liquid phase (interstitial liquid) throughout the length of the column. The left-hand part of the diagram shows the corresponding information for

the adsorbed phase. Each phase consists of four zones: one high in benzene content, one low, one transition zone and one of unused silica gel. The transition zone is short because the H.E.T.S. for a well-packed silica-gel column is low (*4*).

To obtain high recovery of benzene for this system, the liquid phase in Zone 'A,' which is essentially of feed composition, must be subjected to further adsorption. This can be done by displacing the Zone 'A' liquid with a different liquid. When all of the Zone 'A' liquid is so displaced the saturates in Zone 'C' liquid are pushed out of the bed. This second liquid which has been termed push is added in sufficient volume to move the transition Zone 'B' to near the bottom of the bed. The push is a saturate liquid having a different boiling point than the charge to facilitate subsequent separation by distillation.

While push is being added, the effluent from the bed is saturate product. If the flow of push were continued, the aromatics would be gradually desorbed and the effluent stream would be switched from saturate to aromatic product. The flow of push could be continued until the benzene was completely desorbed. However, a more efficient system is to use only sufficient push to displace the transition zone to near the bottom of the bed and then to desorb the benzene with an aromatic stream having a different boiling point from that of the charge. The push, depending on the amount used, replaces some of the adsorbed saturates. The balance of the saturates are displaced by the aromatic desorbent thus permitting subsequent recovery of high-purity benzene. Use of aromatic desorption leaves the gel bed at the end of the cycle filled with aromatic desorbent in both the adsorbed and liquid phases.

The question arises how aromatics can be adsorbed from the feed in the next cycle if the silica-gel bed is full of desorbent aromatics. This is difficult to explain for the actual dynamic system. However, if the situation is taken as a stage-wise operation with the further assumption that all liquid in the pores is adsorbed, the phenomenon is fairly easy to understand. First, it must be realized that aromatics such as benzene and xylene have about the same tendency to be selectively adsorbed. Therefore, the ratio of two aromatics in the adsorbed phase will be substantially the same as their ratio in the liquid phase under equilibrium conditions.

Table 1 shows the calculated composition of both the adsorbed and liquid phases for the assumed top two adjacent theoretical plates. For convenience, all the numbers are parts relative to 100 parts of adsorbed aromatics. This basis gives 130 parts in the liquid phase for this gel. Line 'A' shows both the liquid and adsorbed phases being wholly xylenes from the previous cycle. Line 'B' shows a momentary situation just after feed has displaced the xylenes in the liquid phase. Line 'C' shows that after equilibration some saturates have been adsorbed displacing some xylenes from the adsorbed phase. Since benzene and xylenes have about the same adsorbability, the ratio of these aromatics is

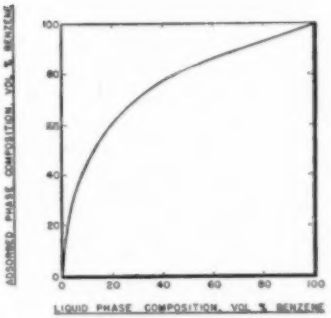



Fig. 2. The adsorption equilibrium diagram for the system benzene-n-heptane.

the same in both the adsorbed and liquid phases. This results in the benzene, as a percentage of benzene plus saturates, being reduced from 30% to about 19% in the liquid phase. Broadly, what has occurred is that benzene has replaced some of the xylenes in the adsorbed phase with the net effect that part of the benzene in the liquid is replaced by xylenes.

The next two lines ('D' and 'E') show the same sort of thing taking place in the next plate down, except that the feed to this plate is the equilibrium liquid composition from the plate above.

## Application to Commercial Concentration

**Benzene and Toluene.** Assume that 1,000 bbl./day of catalytic reformate are available containing about 11% benzene and 16% toluene, or a total of 27% aromatics. The object is to recover the optimum amount of the aromatics at high purity with a minimum use of push, desorbent and silica gel. To do this the gel capacity for feed, the necessary amounts of push and desorbent, and the combined effects of percolation rate, height, diameter and number of silica-gel cases must be considered.

The first problem in designing this unit is to set up the silica-gel bed cycle. Since gels vary slightly, the capacity of any gel for aromatics is established from an equilibrium curve which is determined by permitting fresh silica gel to come to equilibrium with charge stocks having aromatic contents varying up to 80%. In practice it is simpler to use a pure saturate, such as *n*-heptane, mixed with pure benzene and with pure toluene. It has been demonstrated that *n*-heptane approximates the charge saturates sufficiently well for this purpose and that the actual charge containing a mixture of benzene and toluene will lie on a curve intermediate between the points for the two pure compounds. A typical curve arrived at in this manner is shown in Figure 4.

Since the charge stock under consideration contains 27% aromatics, the curve shows that the ultimate gel capacity for aromatics is 0.026 gal. of aromatics/(lb. of silica gel) (cycle). The cycle amount of charge that would result in the entire bed being in equilibrium with the feed is the above figure divided by the fraction aromatics in the charge (0.27), or just under

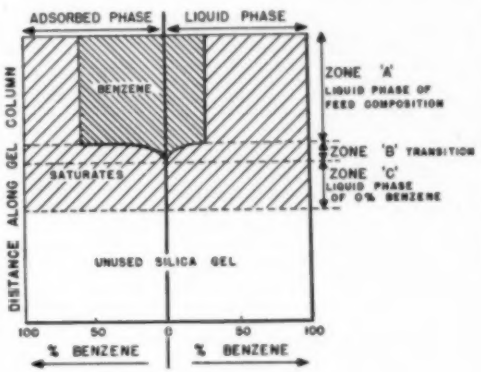



Fig. 3. Schematic composition diagram.

TABLE 1.—CALCULATED COMPOSITION OF ADSORBED AND LIQUID PHASES

| | Adsorbed Phase | | | Liquid Phase | | | |
|---|---|---|---|---|---|---|---|
| | Xylene | Benzene | Saturate | Xylene | Benzene | Saturate | %* |
| | | | Plate Number 1 | | | | |
| A | 100 | .... | .... | 130 | .... | .... | .... |
| B | 100 | .... | .... | ..... | 39 | 91 | 30 |
| C | 57.9 | 22.5 | 19.6 | 42.1 | 16.5 | 71.4 | 18.7 |
| | | | Plate Number 2 | | | | |
| D | 100 | .... | .... | 42.1 | 16.5 | 71.4 | 18.7 |
| E | 76.5 | 8.9 | 14.6 | 65.6 | 7.6 | 56.8 | 13.4 |

* Per cent benzene in benzene plus saturate in liquid phase.
All other values are parts relative to 100 parts of xylene in adsorbed phase.

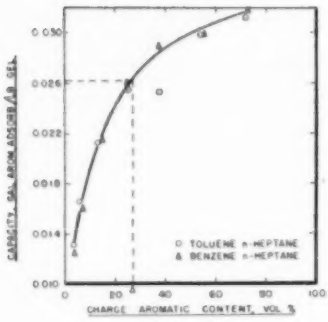



Fig. 4.

one-tenth gal. of feed/(lb. of gel)(cycle). In practice this capacity cannot be utilized because even the bottom of the gel column would have to be in equilibrium with liquid of feed composition. This would result in an incomplete separation of aromatics from saturates because the effluent at this point would be of feed composition. Experience has shown that the actual amount of feed should be 70-80% of the equilibrium value.

Figure 5 shows the aromatic recovery obtained at two aromatic purities plotted against the amount of feed expressed as per cent of equilibrium capacity. These curves were determined by running the charge stock in a cyclic operation at a rate of 0.6 gal./(min.)(sq.ft.) through a column packed with the specific gel to be used. In this work no push liquid was employed. In order to be conservative, a value of 70% is selected which gives 0.068 gal. of charge/(lb. silica gel)(cycle).

The necessary amount of push liquid is determined by making a series of cycles similar to those just discussed, but holding the charge quantity at 70% of the equilibrium value. Figure 6 shows curves for 95 and 97% aromatic purity from such a study with the push liquid varied from 0 to 0.03 gal./(lb. of silica gel)(cycle). In these experiments a percolation rate of 1.5 gal./(min.)(sq.ft.) was used in contrast to the rate of 0.6 previously employed. While the higher rate gives a poorer recovery at any particular purity, it is about the rate employed commercially (as brought out later) and also demonstrates the effectiveness of push more clearly. Because essentially pure aromatic products are required, it would be desirable to have a curve at 99% purity but routine analytical means are not sufficiently accurate. However, since the curve for 95% purity is almost flat and the 97% one shows a pronounced break around 0.01 gal. of push/(lb. of gel)(cycle), it can be inferred that

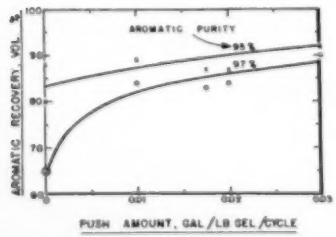



Fig. 6. Curves for 95 and 97 per cent aromatic purity.

TABLE 2.

| | Gallons per Pound of Silica Gel a Cycle | Barrels per Day |
|---|---|---|
| Charge | 0.068 | 1,000 |
| Push | 0.025 | 370 |
| Desorbent | 0.085 | 1,250 |
| Total | 0.178 | 2,620 |

a curve for 99% purity would break in the vicinity of 0.02. Production pilot-plant runs confirm this and show that 0.025 is a safe figure. Butane or pentane are probably the most economical liquids to be used for push in this instance.

For the problem under consideration, mixed xylenes are a good choice of aromatic desorbent since this material is fairly available and easily separated by distillation from the products. Figure 7 illustrates the variation in aromatic recovery with the amount of desorbent liquid per cycle. In these experiments, although no push liquid was used, the level of the curves is again high because the low percolation rate of 0.6 gal./(min.)(sq.ft.) was beneficial.

Assume that a conservative desorbent amount of 0.085 gal./(lb. of silica gel)(cycle) is selected. Recapitulating, the amounts of the various streams are as shown in Table 2.

The result of running these quantities through a silica-gel bed is illustrated by the effluent composition diagram in Figure 8. Almost 100% desorbent is shown at each end of the diagram. If more desorbent had been used the zone of almost pure desorbent at each end of the diagram would have been wider. If appreciably less desorbent, there would not have been a good split between the aromatics and saturates of the charge since Point 'B' corresponds to Point 'A' of the next cycle. The reversal in the desorbent curve between 70 and 80% of cycle reflects charging less than cycle amount, i.e., the bottom of the gel bed contained xylenes from the previous cycle which were displaced before the aromatics from the current cycle appeared.

Assuming sufficient desorbent, it is seen from Figure 8 that the only point at which the saturate and aromatic fractions appreciably overlap is in the push

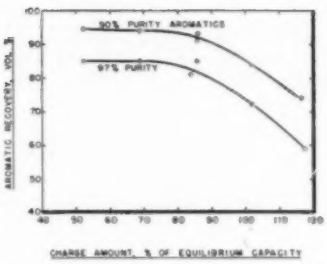



Fig. 5. Aromatic recovery obtained at two aromatic purities plotted against the amount of feed expressed as per cent of equilibrium capacity.

band, the region separated by vertical dashed lines in Figure 8. The recovery of high-purity aromatics can be raised by taking a portion of the cycle shown between the dotted lines and recycling it to the charge stream. Notice that this recycle contains about 30% of total aromatics which is similar to the aromatic content of the charge. Consequently, recycle replaces charge about volume per volume. If less push had been used the aromatic content of the recycle would have been higher and therefore replaced a correspondingly higher quantity of charge.

## Design of Gel Beds

Having found the charge, push and desorbent amounts (for simplicity the influence of recycle is omitted), all the flows to and from the silica-gel cases have been established. Determining the dimensions of the silica-gel beds is strictly a cut-and-try operation with rate, height, diameter, and number of cases being the four factors necessary to consider. Actually, within rather broad limits the final choice of bed dimensions is made largely on the basis of economic considerations.

In any given silica-gel column, increasing the rate of throughput increases the pressure drop proportionately, since operation is always in the

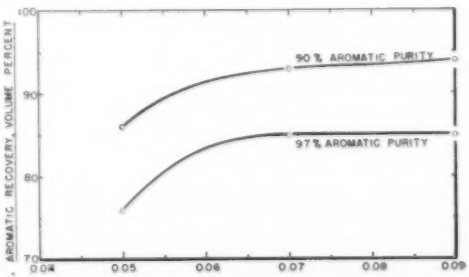



Fig. 7. Variation in aromatic recovery with amount of desorbent liquid per cycle.

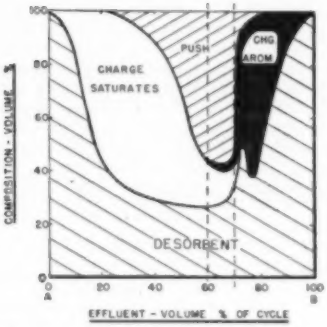



Fig. 8. Typical composition diagram of the effluent during course of one cycle.

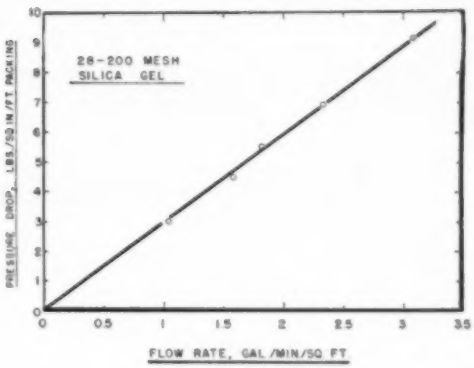



Fig. 9. Relationship of pressure drop to rate for percolation of xylenes.

streamline flow region. Figure 9 shows the relation of pressure drop to rate for the percolation of xylenes through 28-200 mesh gel at room temperature.

The effect of rate must be studied in conjunction with column height. Figure 10 is a plot of aromatic recovery at 95% purity vs. rate for three different column heights, holding diameter constant. Results obtained while charging catalytic gas oil were selected for illustration because this more viscous material emphasizes the effect of rate and height. No push was used but the charge was diluted with an equal quantity of isopentane. As height is increased, the permissible rate for a given recovery is increased. However, at constant rate, pressure drop is directly proportional to height. Therefore, while increased height permits much greater rates of throughput, pressure drop is also increased correspondingly and usually becomes the controlling factor. Practically, height is a safety factor in design. With tall columns any band of mixing between the saturate and aromatic portions is a smaller percentage of the total cycle and the increased cycle time is an added benefit in that the control of this variable is less critical. Everything considered, a height of 15-20 ft. with a rate of 1.5 gal./(min.)(sq.ft.) is a good design basis for most situations.

Initially, fear was expressed that large diameter silica-gel beds might channel and consequently give unsatisfactory separations. This turns out to be the case if adequate distributing and collecting systems are not used and if the bed is not packed homogeneously. However, as shown in Figure 11 a column of 3-ft. diam. and 3-ft. height can be constructed to give results just as satisfactory as a column of 4-in. diam. and 3-ft. height. These runs were made on a petroleum spirits cut containing 16% aromatics. As brought out previously, increased height is beneficial and these columns were deliberately built only 3 ft. high to magnify any possible effect of diameter. Commercial beds of still larger diameter but also of much greater height are expected to give even better performance.

The last selection to be made is the number of cases. With the total 2620 bbl./day of charge, push and desorbent and a rate of 1.5 gal./(min.)(sq.ft.) a gel bed cross section of 51 sq. ft. is required. This area can be obtained in one case approximately 8 ft. in diam., or in three 4½-ft. cases. Since three cases can be staggered to smooth out flows to and from the gel case system, this would be the final design. The cycle time in minutes is calculated by multiplying the total gallons of charge, push and desorbent per pound of gel a cycle by the height of the column and by the number of pounds of gel per cubic foot and dividing by the unit percolation rate. Using 46 lb./cu.ft. as the packed silica-gel density, and a column height of 15 ft. plus the other values previously derived, the cycle time works out to be 82 min.

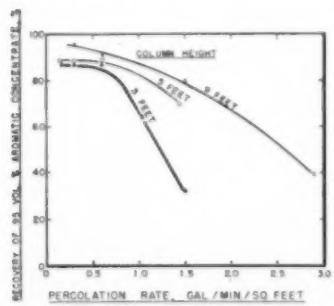



Fig. 10. Plot of aromatic recovery at 95 per cent purity.

## Silica Gel

The three gel cases, 4½ ft. in diam. and 15 ft. high, have a silica-gel capacity of 33,000 lb. The silica gel recommended for the Arosorb process is a gel produced commercially by the Davison Chemical Corp. Through a number of years of cooperative development between Sun and Davison the properties of this gel have been adjusted to produce a highly efficient material.

Small particles of silica gel offer less resistance to adsorption because of shorter pore length. However, a commercial-size case packed with fine silica gel would have an impractically high pressure drop at reasonable flow rates. Particles of relatively uniform size probably would be desirable because the rate of reaching equilibrium for each particle would be more nearly the same, but such particles cannot be packed as densely and larger columns would be required for the same amount of silica gel. Moreover, for a given silica-gel column there would be more liquid held up in the volume between the particles, causing greater mixing between the saturates and aromatics. In addition, silica gel of narrow mesh range is significantly more expensive than broad mesh range material. It has been found that 28-200 mesh silica gel is a good compromise considering the factors of

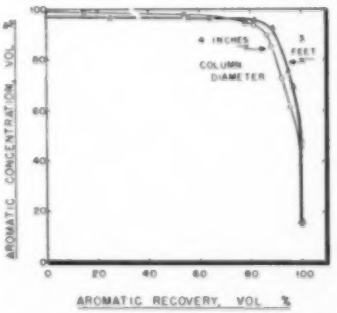



Fig. 11.

efficiency, packed density and cost.

The life of silica gel in the Arosorb process varies with the type of stream being processed and the effectiveness of pretreaters; in general, it is affected by three factors, namely, water, olefins and polar compounds.

Pretreatment driers are necessary to reduce the water content of the feed to the general level of 20 p.p.m. Silica gel has a much greater (*6*) affinity for water than aromatics and according to a published isotherm 20 p.p.m. of water reduces the capacity of silica gel by 15%. This reduction was taken into account in the earlier calculation of silica-gel capacity.

Trace quantities of nitrogen, sulfur and oxygen compounds present in many petroleum fractions are definite gel poisons. Their effect is to reduce the rate at which equilibrium is reached and to reduce the capacity of silica gel. Charge stocks containing readily polymerizable olefins have been found to poison ordinary silica gel. Polymerization of the olefins on the gel surface causes about the same difficulties as organic polar molecules. The Davison Chemical Corp. is working on an olefin-resistant silica gel.

Protecting the silica-gel beds from water and organic polar compounds can readily be accomplished by pretreatment driers of a conventional type. In getting the necessary water reduction, the polar compounds also are reduced to a satisfactory level.

With the above-mentioned type of pretreatment the life of silica gel appears to be considerably in excess of a year, but a life of one year is usually taken for the purpose of economic calculations. Actually, the problem of silica-gel life is more one of the size of the pretreating equipment and the necessary frequency of regenerating the latter. This can be determined only from a thorough experimental study of any particular feed stream.

Because of the current high interest in the recovery of aromatics from catalytic reformates, it is pertinent to mention that these materials are extremely low in gel poisons, and water removal is the only problem.

**Instrumentation.** A multiple number of silica-gel cases is recommended to smooth out the over-all flow of charge and product. A master cycle timer is used to control the length of time each case is on feed, push and desorbent, as well as to keep the cycle of each case staggered in relation to the others. A flow controller on the effluent from each case maintains the rate of flow with sufficient accuracy that the quantities of charge, push and desorbent are substantially proportional to the length of time

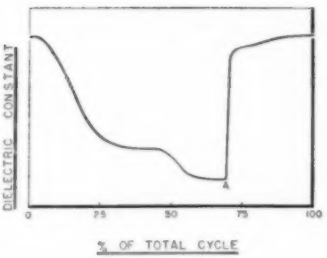



**Fig. 12.**

each of these streams is being fed to the case. If the meter in the case effluent line is of the orifice type, some minor adjustments are necessary to compensate for variations in the viscosity and density of the effluent stream during the cycle.

The effluent is accurately and automatically switched from saturate to recycle to aromatic product lines by an instrument developed in these laboratories. Figure 8 shows a typical composition diagram of the effluent during the course of one cycle. This composition can be followed by dielectric constant which is continuously measured and recorded for each case. Figure 12 is a replot of a segment of the recorder chart. The instrument contains a device which recognizes the reversal in the dielectric constant at Point 'A' and resets the effluent cycle timer accordingly. The latter can be adjusted by means of suitable dials to make the cuts at any selected time intervals thereafter. An instrument of this nature has been operating for some months in the Arosorb pilot plant and is reliable and accurate.

## Summary

If 10% of the case effluent had been recycled, the push and desorbent amounts required for 1,000 bbl./day of fresh feed would have been increased to approximately 500 bbl./day of push and 1,700 bbl./day of desorbent. Recycle would have increased the aromatic recovery from 85% to 95% or higher and insured the production of nitration grade benzene and toluene. A light acid treatment usually is required to meet acid wash specification, but the Arosorb process tends to reject olefins to the saturate product thereby minimizing acid treatment of the aromatics.

Breadth and level of boiling range or aromatic content of charge stocks do not limit the application of the process. The situation discussed here is one of the most complicated because essentially pure compounds are produced. When a stream is merely being processed for high aromatic solvents, high cetane Diesel fuel, or similar stocks not requiring high purity, the process can be materially simplified. Push need not be employed and therefore two distillation columns are eliminated. However, the installation of the more complicated plant gives the refiner a wider choice in the types of stocks he may process and the quality of products produced.

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## Discussion

**D. I. Saletan** (Shell Chemical Corp., Martinez, Calif.): In conventional distillation processes there is generally a calculable minimum quantity of energy required for the separation and I assume in this sort of intermittent flow process that the heats of desorption and adsorption are involved. Would you make a few comments on the thermal effects?

**J. I. Harper:** The actual temperature range that the gel bed goes through is only a few degrees.

**Anonymous:** It might help some of us if you would expound or expand on how you get the xylene out of the bed. You left the silica gel wet with xylenes, and you didn't explain how you got the xylene out.

**J. I. Harper:** That is a most complicated thing to explain. I think I can give you an idea though. Let's consider the bed full of xylene in both the liquid and adsorbed phase as it would be at the end of the cycle. Then put in a charge stock containing benzene and heptane. Now the heptane doesn't tend to adsorb to any great extent, but the benzene and xylene have about the same adsorbability. What happens is that some of the benzene replaces some of the xylene in the adsorbed phase and some of the xylene replaces some of the benzene in the liquid phase thus reducing the benzene content of the liquid. Consider first that this has occurred in the top layer of gel. As the liquid passes down through the bed the same thing happens again and again until all the benzene in the liquid phase is replaced with xylene. That, of course, is a loose picture of the operation but explains how it works.

(*Presented at A.I.Ch.E. Forty-fourth Annual Meeting, Atlantic City, N. J.*)

# THE ROLE OF SUPER-REFRACTORIES

## *In the Chemical and Hydrocarbon Industries*

A. A. TURNER, H. M. KILLMAR, and R. W. BROWN

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SUPER-REFRACTORY materials of construction are being used in large quantities as important components of high-temperature chemical-process apparatus. They embrace ceramic and self-bonded, kiln-fired products made from silicon carbide, fused alumina, and synthetic electric-furnace mullite, as well as electrically fused-cast refractories, which, as the name implies, are made by melting carefully selected ingredients in electric furnaces, casting the melt into shape, and annealing the casting to produce the completed piece.

The selected materials forming the major parts of these products offer unusual properties which, through the use of special manufacturing techniques, are retained to the highest degree in the finished refractories. To distinguish super-refractories from refractories produced directly from natural clays and ores, the term electric furnace refractories often is used.

### Classification

The terms silicon carbide, fused-aluminum-oxide and electric-furnace-mullite refractories and fusion-cast high-alumina materials classify super-refractories by types. Within each family type, many specific varieties are available, each of them filling distinct needs of industry. While it is true that the properties of the bonded materials are in a large measure a function of the basic ingredient, it is not generally realized by some process engineers that the make-up of the ceramic bond, often a very small part of the refractory, also greatly influences the behavior of the refractory in service. These bonds, as well as other factors, give rise to the specific varieties of bonded refractories, whereas the varieties of the homogeneous fused-cast materials result from the constituents of the melts used in their manufacture.

Table 1 lists several types of super-refractory materials and shows the chemical compositions and some of the physical properties of specific widely used varieties of each type. Unfortunately, these data can serve only as rough guides in the selection of a material for use. The choice of the proper types of material and the proper variety is often difficult. While super-refractories are necessary for very high-temperature service, many critical applications do not depend on high refractoriness, except possibly to provide a margin of safety in operation. Most often the reason for their selection is some other unique property or combination of properties.

Frequently processes are commercially feasible only because satisfactory special refractories are available as construction materials. By making full use of the properties displayed by super-refractories, new processing techniques have been made available to the chemical-plant operator. Meticulous attention to the selection of the proper material, however, is essential for best results. Close cooperation between super-refractory manufacturers and the chemical industry has resulted in many pieces of highly efficient equipment and has made possible improved performance from older long-used pieces of apparatus.

**Silicon Carbide.** The most versatile and widely used type of super-refractory material today is bonded silicon carbide, and it is logical that its use is being greatly extended in the process industries as the trend to high temperatures and indirect heating continues. Writers have discussed the role played by this material in the chemical and metallurgical industries for muffles in muriatic-acid furnaces and multiple-hearth roasters and for externally heated retorts, recuperator tubes, still settings, and metallurgical furnaces (*9*). Its use in the manufactured-gas utility industry for water-gas-generator linings, flue-connector linings, and carburetor linings is well understood (*8, 12, 16, 17, 21*). Bonded silicon carbide has long been used for gas-fired radiant tubes, which are employed where combustion gases must be kept away from the work and where special atmospheres are used for heat-treatment. Additional basic research has recently been completed providing engineers and designers with much-needed data on the tube heat-transfer rates, maximum permissible temperature of use, and allowable heat-release rates inside such tubes (*2*). Early work with externally heated tubes for the reforming of hydrocarbons produced much of the foundation for present-day interest in the regenerative reforming process (*11*). Silicon carbide grain is employed as a major ingredient in retorts used in the zinc industry in the Belgian retort method of reduction (*5, 10*); however, this process has been largely superseded in the past decade by the vertical-retort method, which employs heat-transfer walls of silicon carbide. Linings of silicon carbide are used in vapor off-takes and condensers in a new process based on the electrothermic reduction of zinc ore (*27*) and for the trays in the process of zinc refining by distillation (*22*). Special muffle processes for the production of zinc oxide and zinc depend upon the unique properties of this material (*15*).

### New Processes

There are many relatively new high-temperature processes in which silicon carbide and other types of super-refractories are important components of production equipment. The use of silicon carbide for regenerator checkers is a case in point. The properties of excellent thermal conductivity and high emissivity, coupled with high density and

TABLE 1

TYPICAL CHEMICAL ANALYSES AND PHYSICAL PROPERTIES OF BONDED AND FUSED CAST REFRACTORIES MADE FROM ELECTRIC FURNACE PRODUCTS

| | Grade A Silicon Carbide (A) | Special Silicon Carbide | Fused Alumina | | Lightweight Fused Alumina | Electric Furnace Mullite | Beta Alumina Fused Cast | Alpha-Beta Alumina Fused Cast | Alumina-Chromite Fused Cast |
|---|---|---|---|---|---|---|---|---|---|
| | | | Low Silica | Regular | | | | | |
| **Chemical Analyses** | | | | | | | | | |
| Ignition | 1.37 | ..... | Nil | 0.15 | Nil | Nil | 0.18 | 0.07 | 0.08 |
| $SiO_2$ | 89.16 | ..... | 0.39 | 0.05 | 0.05 | 75.53 | 0.06 | ..... | ..... |
| $Al_2O_3$ | 7.75 | ..... | 99.30 | 99.55 | 81.02 | 23.47 | 98.80 | 94.56 | 79.16 |
| $Fe_2O_3$ | 0.87 | ..... | 0.50 | 0.75 | 16.80 | 0.27 | 0.50 | 0.50 | 1.06 |
| CaO | 0.87 | ..... | 0.13 | 0.27 | 0.09 | 0.27 | 0.11 | 0.12 | 4.09 |
| MgO | <0.10 | ..... | Nil | 0.10 | Tr | <0.10 | <0.10 | 0.23 | 0.25 |
| $Na_2O$ | ..... | ..... | Tr | 0.10 | 0.10 | <0.10 | <0.10 | <0.10 | 4.23 |
| $TiO_2$ | ..... | ..... | 0.37 | 0.53 | 1.59 | 0.40 | 5.62 | 4.07 | 1.09 |
| $Cr_2O_3$ | ..... | ..... | 0.01 | 0.07 | .40 | 0.09 | <0.05 | <0.05 | 1.39 |
| | ..... | ..... | ..... | ..... | ..... | ..... | ..... | ..... | 8.77 |
| **Physical Properties** | | | | | | | | | |
| Pyrometric Cone Equivalent | 38 | ..... | 39–40 | 38–39 | 38–39 | 38–39 | 39–40 | 39–40 | 38–39 |
| Weight of 9" × 4½" × 2½" Straight in Lbs. | 9.2 | 10.3 | 10.4 | 10.3 | 4.8 | 9.0 | 10.2 | 11.6 | 11.6 |
| Average Bulk Density C.G.S. | 2.55 | 2.87 | 2.90 | 2.87 | 1.30 | 2.50 | 2.80 | 3.29 | 3.20 |
| Porosity—% | 13.2 | 7.9 | 21.9 | 22.6 | 66.6 | 22.7 | 7.5 | 2.5 | 4.5 |
| Mean Specific Heat 0° to 1400°C | ..... | ..... | ..... | ..... | 320 | 230 | ..... | ..... | ..... |
| Mean Coefficient of Exp. per °C. 25° 1400°C | $4.4 \times 10^{-6}$ | $4.4 \times 10^{-6}$ | $7.4 \times 10^{-6}$ | $7.4 \times 10^{-6}$ | $8.6 \times 10^{-6}$ | $5.9 \times 10^{-6}$ | $8.6 \times 10^{-6}$ | $8.2 \times 10^{-6}$ | $8.6 \times 10^{-6}$ |
| Thermal Conductivity at 2200°F in British Units | 109 | 113.5 | 24 | 24 | 7 | 15 | 24 | 31 | 31 |
| Modulus of Rupture @ 1350°C | 2000 | 5640 | 200 | 658 | 63 | 433 | ..... | ..... | ..... |
| 1500°C Load Test @ 25 lb./Sq.In.——Contraction Cold | Nil | 0.0% | 0.50% | 0.35% | 0.65% (B) | 0.15% | Nil | Nil | Nil |
| Abrasion Resistance | Excellent | Excellent | Fair | Fair | Poor | Fair | Fair | Good | Excellent |
| Relative Resistance to Spalling | High | Very High | Good | Good | Good | Very Good | Fair | Low | Low |

(A) This covers a variety of silicon carbide refractory commonly used. Where required, higher SiC content varieties can be supplied.

(B) @ 12.5 lb./Sq. In.

refractoriness, make bonded silicon carbide an ideal heat-storage medium for elevated-temperature operations. For any given temperature, the total amount of heat that can be stored in a regenerator is primarily a function of the weight of the materials used and their specific heats; however, the rate at which heat can be absorbed and discharged is a function of the thermal conductivity and emissivity of the materials used and the cycle times employed. It has been shown that the behavior of refractory materials as heat-storage mediums for regenerators can be compared mathematically (*14*), and the relative effectiveness of bonded silicon carbide brick and fireclay brick is illustrated in Figure 1. These curves graphically show why the use of a material having a thermal conductivity roughly 10 to 11 times that of fireclay will result in a checker setting that will operate faster and deliver more heat and reabsorb more heat per unit of checker volume and time than will a regenerator in which a low conductivity material is used. In other words, silicon carbide checkers make more heat available per unit of time and of volume of apparatus.

**Oil-Gas Equipment.** Low B.t.u. oil gas for city distribution is produced from heavy oil by deep thermal cracking. Generator carbon black is a by-product of this process. The preheated oil is sprayed upon hot checkers in a regenerative machine consisting of large checker settings enclosed in refractory-lined steel shells. The checkers are heated by oil burners with additional heat being supplied by the burning of that portion of the carbon black deposited in the settings during the make-run. The heat stored by the checkers in the heating part of the cycle is used to vaporize and crack the oil fed to the machine during the cracking part of the cycle.

With the low-thermal-conductivity checker materials formerly used, long cycle times were necessary. To obtain additional available heat-storage capacity, silicon carbide checkers have now been substituted for clay, usually in only the top four courses of the setting. The air-blast rates also have been increased. These changes made possible a 50% reduction in cycle times after the sets were rebalanced for best results, and gas-making capacity has been approximately doubled. Moreover, the by-product carbon deposited in the checkers and collected in the wash boxes has been reduced in proportion.

For the same reasons, silicon carbide checkers have been used in certain sections of the checker settings in carburetors and superheaters of water-gas machines and have resulted in improved oil-cracking capacity and efficiency. They are used in the igniters of hydrogen generators where the additional available heat-storage capacity gives improved operation.

Several new designs of oil-gas generating equipment have recently been developed and are now widely used to produce high B.t.u. gases from light and heavy oils. Such gases are being manufactured by utilities to serve as supplements to and substitutes for natural gas.

Silicon carbide checker brick is now considered standard for four or five courses in the generating shells of the "twin-generator" process operating on light oil or gas oil when these machines are operated by companies that produce appreciable quantities of oil gas for peak shaving and stand-by (*7*). The additional usable heat-storage capacity of the super-refractory results in improved oil-cracking capacity and efficiency; cold machines can be brought to working temperature and can produce quality gas sooner, and the machine responds more readily to operating adjustments and controls.

Large quantities of oil gas can be produced more economically from cheaper heavy-residuum oils than from the more expensive lighter oils. To handle the former and take advantage of their lower cost, a regenerative system known as the Hall high B.t.u. oil-gas process has been developed and is being rapidly adopted by utilities (*1, 13*). The process is cyclical. Four distinct checker settings are used, two serving as superheating or fixing chambers and two working as oil-vaporizing and cracking units. The latter two are heated by auxiliary oil burners and by the combustion of carbon from two sources. The first of these is the Conradson carbon from the make-oil, and the second is the carbon deposited on the checkers from the cracking of the oil-gas vapors. In the process equipment, silicon carbide brick is almost universally used in the generating shells for the major portion of the checker settings, as illustrated in Figure 2. Figure 3 illustrates a two-shell Hall process machine in which the gas-generating checker settings of silicon carbide have been superposed above the superheating or fixing settings. Not only does the use of the two large shells reduce investment costs, but also these machines take up less floor space.

Another piece of oil-gas generating equipment is illustrated in Figure 4. The process incorporates the principle of counterflow between the carrier gas and oil fog in the reactor, which is an empty chamber lined with silicon carbide brick to release maximum heat for the efficient cracking of the oil. Regenerators are used for preheating the air and steam, which makes for more efficient operation. This same refractory is used in the process industry to make oil gas from which butadiene is recovered for synthetic-rubber production.

**Service.** The silicon carbide brick used as checkers in all the equipment described here has a finite life, and the total service obtained is dependent upon local conditions. The brick gradually fails by disintegration due to thermal shock. In the machine using an empty reactor, only one face of a brick in the heat-storage lining is exposed to the oil spray, and the life is increased. Results have shown that checker service is a function of the temperature of cracking, the type of oil used, the shape of the individual checkers, and the variety of silicon carbide super-refractory employed. Research work followed by field trials on the problem of silicon carbide checker-setting life has proved that soap brick is superior to straights and to all other standard brick shapes in cracking resistance and that some mix varieties give longer life regardless of shape. Certain special shapes are better than soaps; for instance, small solid cylinders are extremely resistant to breakup.

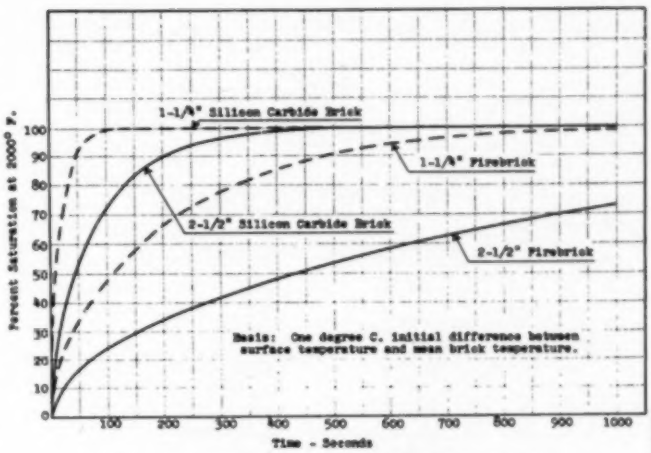


Fig. 1. Heat saturation curves—silicon carbide brick vs. firebrick.

Unique crack-resistant checker shapes can be economically made and give promise of superior performance in current trials.

It should be emphasized that in the checker applications mentioned in this paper, a relatively expensive material is in large-scale commercial use, not because the checker settings last for a proportionately longer time than lower priced refractories, but because their use makes possible more efficient and economical operation at higher set capacities. It is seldom, if ever, possible to obtain satisfactory results by simply making a direct substitution of silicon carbide checkers for clay checkers; the equipment in which the checkers are used must be rebalanced and the cycles readjusted to take advantage of the larger available heat-storage capacity provided (*see* Figure 1).

**Regenerative Reforming Apparatus.** Closely allied with the foregoing is the current commercial use of specific varieties of bonded silicon carbide checkers in regenerative chambers used for the thermal reforming and thermal-catalytic reforming of various hydrocarbons. Because of the unusual properties of

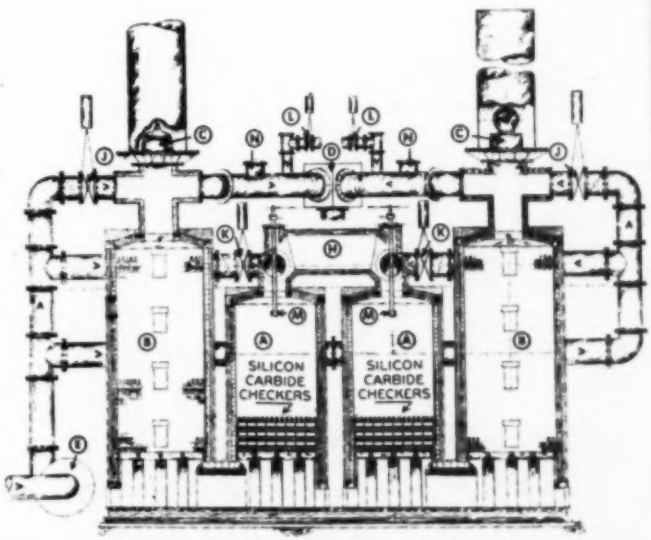


Fig. 2. Four-shell regenerative heavy oil gas machine.

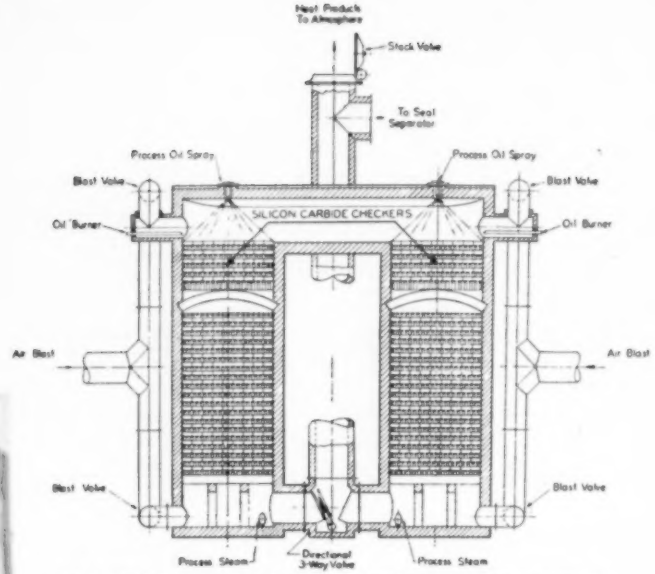


Fig. 3. Two-shell regenerative oil-gas set. Universal operation—light or heavy oils.

silicon carbide, smaller regenerative chambers can be used, cycle times can be short, and the temperature swings can be controlled within narrow limits for maximum yields.

In certain areas of similar types of equipment operating under extremely high-temperature conditions coupled with destructive atmosphere attack, refractory and inert fused-alumina checkers are longer-lasting than those made from silicon carbide. Checker settings made of a combination of fused-alumina brick and silicon carbide brick have been successfully used, the desirable properties of each refractory being thus utilized in specific regions of the regenerative setting.

A newly developed reforming process that has possibilities of wide application in the hydrocarbon-processing industry to date has used specific varieties of bonded fused aluminum oxide because of the density and chemical stability of this material. The lower conductivity of fused alumina, compared with silicon carbide, is largely offset by certain design features. Twin regenerative settings of thin aluminum oxide checker tile, which when put in place form a multiplicity of continuous gas passages within the heat-storage medium, form the heart of the apparatus. These settings permit close control of residence times. Cracking stock with steam and air is reformed in one-half of the furnace, quenching is accomplished in the setting making up the other half of the furnace, and the sensible heat removed from the reformed gases is consumed in the endothermic reactions of reforming when the cycle is reversed, without the need of auxiliary heat.

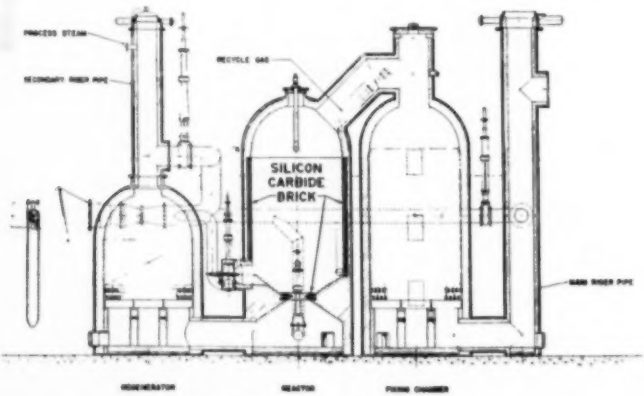


Fig. 4. Regenerative reverse flow oil gas process machine.

Such equipment may be used to reform natural gas and other hydrocarbons. Its use is contemplated for the production of unsaturated hydrocarbons for organic chemical synthesis, for the manufacture of mixtures of hydrogen and carbon monoxide for catalytic synthesis, and for hydrogen generation.

**Continuous Reforming Apparatus.** Special manufacturing techniques have been devised to produce silicon carbide cylinders with remarkable uniformity and excellent chemical stability. The technique of joining sections of refractory tubes into continuous units 25 to 30 ft. long has been highly developed by a process-equipment designer. A piece of apparatus has been developed for operation at high-temperature levels to perform a wide variety of operations (*3, 20*). Because the process is continuous and gas flow is in one direction only, the usual reversing valves and controls are eliminated. Figure 5 illustrates the features of design. Four super-refractory materials are used in the furnace. The silicon carbide tubes serve as externally heated high-temperature heat-transfer media. The excellent hot strength of this material permits combustion-chamber temperatures of 2500 to 2600° F.; therefore, operations can be carried out in a range above that possible with alloy tubes. The high temperature of operation and the excellent thermal conductivity of the tubes permit heat deliveries in excess of those obtainable with other equipment. The bottom short sections of the vertical tubes are made of a completely new type of silicon carbide that is highly resistant to failure resulting from thermal stresses. Corebreakers, which may be catalyst impregnated, of high-purity bonded-electric-furnace mullite sometimes are placed inside the tubes.

The furnace is divided into zones to facilitate close temperature control by electric-furnace-mullite domes; these also serve to keep the sections of tubes in vertical alignment and to baffle the flow of hot combustion gases for maximum convection-heat transfer. The tubes are constructed with patented ground ball-and-socket joints and are effectively sealed with a super-refractory cement. Operation of the unit is simple because it is continuous. The silicon carbide tubes are heated externally by tangentially fired burners arranged vertically in the furnace. The burners in individual zones are operated by separate temperature controls. Products of combustion are forced down through the central portion of the furnace through center openings in the mullite domes and into the waste-heat flue located in the bottom zone. Feed stocks are introduced at the top of the unit into the silicon carbide tubes and pass downward through the 28-ft. tube assembly into a common collecting or quenching chamber. The product from this chamber then travels through a train of processing or cooling apparatus to its point of final use.

This equipment can be used for continuous reforming of hydrocarbon feed stocks. Gases with controllable-flame characteristics desirable for various chemical and metallurgical processes can be economically produced in these

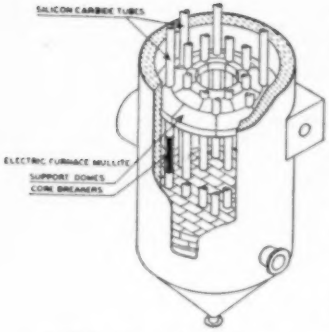


Fig. 5. Continuous reforming furnace.

units. Commercial furnaces containing up to 14 cracking tubes are under erection. Equipment of similar design can be adapted for the continuous superheating of steam, air, or other gases.

**Synthesis Gas Reactors.** Many types of refractories are playing vital roles in the commercial and experimental apparatus used for the manufacture of synthesis gases from oxygen, steam, and hydrocarbons, either pulverized coal or natural gas. In continuous reactors operating at high temperatures with pulverized coal as fuel, the materials of construction must be refractory and chemically stable and must resist the corrosion of molten coal ash relatively high in iron. Ordinary materials dissolve and fail rapidly. One variety of fusion-cast high-alumina refractory displays remarkable resistance to slag attack. This material contains approximately 80% of alumina and is made by fusing alumina with a lesser proportion of chromite.

Microscopic examinations show that the primary crystal phase is a solid solution of alumina and chromic oxide with a ground mass of high-alumina spinel. No interstitial glass is observed (*19*). Following successful small-scale tests, this material is giving excellent results as the reactor lining in experimental generators using a fairly high-ash coal, and commercial plants are being designed around its use. Basic research on synthesis-gas production has been done by the Bureau of Mines with various varieties of coals. Bonded silicon carbide reactor linings have given satisfactory results (*25, 26*); however, it is believed that the corrosion resistance of the fused-cast high-alumina material makes it superior for full-scale, plant-size equipment.

In these synthesis-gas-generating units, bonded refractories made from essentially hollow spheres of fused alumina have also played vital roles as refractory components of the apparatus. Brick or shapes made from this material retain the inertness and refractoriness of fused alumina to a marked degree; because of the sealed pores produced by the hollow spheres, the refractories are light in weight and have low heat conductivity and low heat capacity. These properties make an excellent high-temperature insulating material, and in the synthesis-gas reactor operating on coal it is used as the high-temperature insulating material behind the fused-cast high-alumina corrosion-resistant lining. Similarly, it is used to back up the fired periclase brick in pebble stoves used to superheat steam for the synthesis-gas generator operated by the Bureau of Mines. These applications exemplify the rather general use of this refractory in combination with other super-refractories for very high-temperature work. In this way composite refractory structures can be used with the result that many times thinner, less expensive linings of super-refractories are necessary.

This light-weight, fused-alumina material is, of course, used independently of other special refractories. In the chemical industry it is employed as linings in direct-fired air heaters and as combustion chambers fired with clean fuels. In these and other installations it serves simultaneously as a super-refractory lining and as insulating material without the use of other refractories.

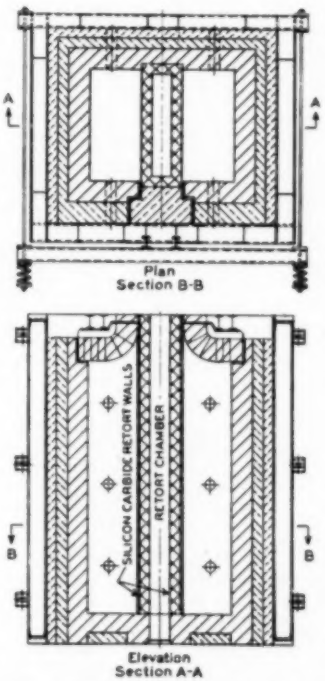


Fig. 6. General arrangement of continuous vertical retort.

## Fusion Apparatus

New processes that involve the fusion of chemical raw materials are being developed. For example, electric furnaces and regenerative fuel-fired tank furnaces are being employed for the melting and processing of phosphate rock. When regular refractories are employed for the lining of the tank, they fail rapidly, owing to corrosion by the melt at high temperatures; therefore corrosion-resistant, fused-cast products are being used to give satisfactory tank life. The quality of the melt is thus improved because their slow solution rate, unlike that of clay materials, holds to a minimum the content of dissolved refractory in the melted product.

Special refractories have been used in the trouble-spot areas of equipment for electrochemistry. The varieties of fused-cast high-alumina materials composed almost entirely of beta alumina (*23*) are used for the linings of the cells producing magnesium by the electrolysis of fused magnesium chloride (*24*). In these cells, the material must stand the attack at the metal line of molten magnesium, chlorine, and fused electrolyte and must resist the thermal stresses usually involved at the top of a molten bath.

**Vertical Retorts.** The successful use for many years of externally heated silicon carbide vertical retorts for the reduction of zinc ore is well known (*6*). Similar units using end compression on the retort walls for maximum joint tightness have been used for many years to calcine charcoal and to reduce cobalt and chrome ores. The heat-transfer walls can be 25 to 30 ft. tall and up to 6 ft. long. The width of the slot is dependent upon the material being treated. These retorts operate continuously, a steady flow of material entering at the top and the treated material being discharged at the bottom.

High-temperature retorts of similar design are now being successfully operated on pilot-plant scale in a number of confidential developments. Retorts are also being built for the calcination of briquetted charges of green petroleum coke and other varieties of coal and petroleum-coke charges. Such equipment is also being used for fluidized-bed work. A typical calciner is illustrated in Figure 6. New retorts are being erected for additional basic research on calcination and coking. Successful operation of these retort calciners is directly dependent on the use of carefully selected super-refractory materials made from electric-furnace products.

**New Flat Coking Ovens.** There are today many closely related factors contributing to a critical shortage in important types of industrial carbons (*18*); however, process-equipment designers are developing apparatus to produce certain types of industrial carbons from petroleum residues and other by-products.

Super-refractories make possible effective use of flat coking ovens suitable

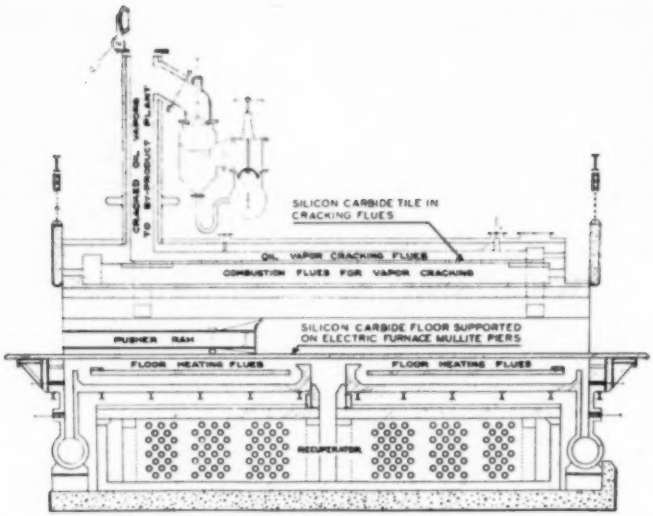
CRACKED OIL VAPORS TO BY-PRODUCT PLANT
SILICON CARBIDE TILE IN CRACKING FLUES
OIL VAPOR CRACKING FLUES
COMBUSTION FLUES FOR VAPOR CRACKING
PUSHER RAM
SILICON CARBIDE FLOOR SUPPORTED ON ELECTRIC FURNACE MULLITE PIERS
FLOOR HEATING FLUES
FLOOR HEATING FLUES
RECUPERATOR

Fig. 7. Typical design of flat oven.

for the carbonizing of tars, pitches, acid sludges, and other carbonaceous materials from chemical operations. Such ovens can be used for treatment of both high- and low-volatile coals to produce highly reactive specification coke for chemical raw materials. A certain type of flat oven is being operated to make electrode coke, and, in contrast to other equipment in which up to one-half of the green petroleum coke charged is consumed as fuel, this oven uses cheap oil or gas fuel, and a high yield of quality coke is recovered.

Figure 7 is a vertical cross section through a recent design of broad or flat oven designed for coking petroleum residues to produce electrode coke. The oven consists of an underfired silicon carbide refractory floor supported on bonded-electric-furnace-mullite piers. The oven may be built with integral cracking flues, to give a thermal treatment to the oil vapors for the maximum yield of aromatics, as is shown in the figure, or may consist simply of an underheated floor, the volatiles being drawn off for further treatment in separate apparatus. These structures are relatively inexpensive and simple in design, and their importance for producing coke for electrode manufacture during the present emergency cannot be overemphasized.

**Pebble Heaters.** Pebble heaters, operating at high temperatures, are relatively new pieces of processing apparatus. They are used for a variety of thermal-treatment work in industry. They can be used to superheat air and steam and do thermal-reforming work. Their primary advantage is that a continuous flow of relatively-constant-temperature product is obtainable. Because of the critical high temperatures involved and the combination of destructive atmospheres and abrasion in certain locations in the units, the materials used must be special refractories. In one such unit, the temperatures of operation caused slow but steady slumping and squashing of the fireclay materials originally specified. Fused aluminum oxide substituted for these materials in the same service cracked and shrank. Excellent results are now being obtained with electric-furnace mullite. Length of campaigns has approximately doubled, and slumping is negligible. Moreover, the unit can be run at higher temperatures to give a higher temperature product. This electric-furnace-mullite-base refractory has been used in other pebble-heater combustion chambers for some years. Another heater unit used for hydrocarbon reforming employs a variety of super-refractories and serves to illustrate the roles played by a number of special products. In this heater the moving bed of bonded-high-alumina pellets is supported in the high-temperature heating chamber on chunks or lumps of electrically fused aluminum oxide and bonded mullite which are, in turn, held in place by a perforated dome of bonded silicon carbide. Because of its chemical stability and inertness, bonded mullite is used for the dome skewbacks. Silicon carbide tubes are used in critical sections to resist the abrasion of the moving hot-pebble stream.

By substituting a material to be treated for the usual heat-exchange pellets, pebble heaters can be operated as calciners. In one instance, in a heater calcining brucite, bonded aluminum oxide materials make up the combustion chamber and calciner tube linings. Aluminum oxide refractories are used in this application because of their refractoriness and chemical compatibility with the raw material treated.

**Abrasion-resistant Equipment.** Use of bonded materials made from electric-furnace products or fused-cast products as abrasion-resistant materials of construction has displayed the marked versatility of these products. The resistance to wear of bonded silicon carbide is to be expected in the light of its prominence in the abrasive industry. Fusion-cast products have the added advantage resulting from their homogeneity and lack of relatively soft bonds (*4*). Use is made of both of these materials in cyclone-dust-collector cones, coke chutes and hoppers, ash hoppers, linings of high-velocity-gas mains, and in newly developed gas scrubbers. These materials, moreover, are capable of resistance to the attack of abrasion coupled with corrosion. As linings in the piping to and from sulfur dioxide converters in sulfuric-acid plants, bonded silicon carbide materials give trouble-free service.

## Newer Refractory Materials

An interesting new material is now available to equipment designers. Refractory, corrosion-resistant wool or fiber is being produced for high-temperature insulation and for packings in high-temperature-furnace expansion joints and similar locations. Unlike glass fiber, this material contains only traces of alkalies; therefore, it is of interest for the filtration of corrosive liquids and gases both hot and cold. It is much more refractory than glass fiber and does not devitrify below 2300° F.

Inert catalyst carriers, for fixed- and fluidized-bed operations, are being used in the form of porous aggregates and rugged porous pellets and grains made from silicon carbide and fused aluminum oxide.

Porous, ceramically bonded fused-alumina tile and tubes are used for filtration of many liquids and gases and as the support for other filter materials. In addition, these porous mediums are employed for gaseous-diffusion purposes.

It should be emphasized again that the best service is obtained from these products when they are applied with engineering skill. Super-refractory manufacturers are in a position to consult with prospective users and, through such cooperation, misapplications that have been made in the past can be avoided. Within our knowledge there have been cases where promising processes have been abandoned as the result of inadequate attention being given to the refractories employed or to details of construction or operation as they may affect the refractories selected. As demonstrated by the achievements to date, with close cooperation between user and producer, the role of super-refractories in the chemical industry will assume ever-increasing importance.

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# MULTICOMPONENT DISTILLATION

## IV — *Determination of Minimum Reflux*

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**Two alternate equations for the minimum reflux ratio are shown. Constant molal reflux is assumed. Both equations are exact when the volatility ratios are constant throughout the column. The first equation is designated the over-all balance equation, and is an analytical expression of the method originally proposed by Underwood (*14*); the second is designated the pinch balance equation and is an analytical analogue of the Colburn correlation (*3*). A logical means of determining the equivalent constant $\alpha$ values for use in the over-all balance equation is given. When the keys are adjacent, either the over-all balance equation or the pinch balance may be used to compute minimum reflux. For a system containing any number of split keys, the over-all balance equation is recommended.**

THE beginning of the theoretical study of minimum reflux for multicomponent systems is marked by the derivation of two simple equations, by Fenske (*4*) and Underwood (*12*). Although these equations were not adequate, except under restricted conditions, for the accurate determination of minimum reflux, they supplied a foundation upon which later workers have built. Using these equations and also introducing assumptions since found to be rough approximations to the true situation, various methods have been proposed for solving the minimum reflux problem (*2, 5, 6, 11*). It remained for Jenny (*8*) to clarify and define the exact conditions existing in a column at minimum reflux. Taking these conditions into account, Colburn (*3*) developed a widely used empirical correlation for the calculation of this reflux. From an analytical point of view, one of the outstanding developments in the theory was the algebraic method of calculation given by Underwood (*13-15*).

In the present paper, the determination of minimum reflux is approached from two points of view:

1. For systems in which the relative volatilities remain constant throughout the column, equations analogous to Underwood's, but carried analytically a step further, are developed.
2. For systems in which the relative volatilities may vary, an equation similar in form to that of Colburn's correlation is shown. Consideration of the latter equation provides a logical basis for the choice of relative volatilities for use in the Underwood-type equations. These equations will hereafter be referred to as the pinch balance equation (theoretical analogue of Colburn's empirical formula) and over-

Multicomponent Distillation, Part I, *Constant Volatility Ratio and Constant Reflux*, appeared in November, 1948, page 855. Part II, *Application of Algebraic Equations to Doublet Separations*, in January, 1950, page 36, and Part III, *Equations in Product Form and Simplified Application*, in May, 1952, page 254.

Original manuscript in its entirety, including details of derivations upon which paper is based, is on file (Document 3597) with the American Documentation Institute, 1719 N Street, Northwest, Washington, D. C. Microfilm obtainable by remitting $1.00 and photocopies by remitting $4.35.

all balance equation (Underwood's equation generalized analytically to include split keys).

**Conditions at Minimum Reflux.** The minimum reflux ratio at which specified separations of two key components can be carried out is the reflux ratio for which an infinity of plates in each section of the column is required. When the reflux is at this value, there is in each section a region (called the pinch) extending over an infinity of plates, within which the composition change from plate to plate is infinitesimal. At minimum reflux, no components heavier than the heavy key appear in the top product, and no components lighter than the light key appear in the bottom product.

The basic equations from which the over-all and pinch balance equations are derived are as follows:

$$\Omega_r(\phi) \equiv \sum_{j=1}^{c} \frac{Dx_{Dj}}{1-\frac{\phi}{a_{prj}}} - V_r \tag{1}$$

$$\Omega_s(\phi) \equiv \sum_{j=1}^{c} \frac{Bx_{Bj}}{1-\frac{\phi}{a_{psj}}} + V_s \tag{2}$$

where the subscripts $pr$ and $ps$ refer to the rectifying and stripping pinches, respectively. The important quantities $\phi_{rj}$ and $\phi_{sj}(j = 1 \text{ to } c)$ are defined by the equations $\Omega_r(\phi_{rj}) = 0$ and $\Omega_s(\phi_{sj}) = 0$.

The vapor and liquid compositions in the pinches are

$$y_{pri} = \frac{(1-\rho)x_{Di}}{1-\frac{\phi_{rh}}{a_{pri}}}; \quad x_{pri} = \frac{\phi_{rh}}{\rho a_{pri}} y_{pri}$$

$$y_{psi} = \frac{\left(\frac{1-\sigma}{\sigma}\right)x_{Bi}}{1-\frac{\phi_{si}}{a_{psi}}}; \quad x_{psi} = \frac{\sigma\phi_{si}}{a_{psi}} y_{psi}$$

When the relative volatilities remain constant throughout the column, it is convenient to make use of the quantities $\phi_{fj}(j = 1 \text{ to } c)$, where $\phi_{fj}$ is defined by the equation $\Omega_f(\phi_{fj}) = 0$, with

$$\Omega_f(\phi) \equiv \sum_{j=1}^{c} \frac{X_{fj}}{1-\frac{\phi}{a_j}} - (1-q) \tag{3}$$

It was shown in the original manuscript for this paper that when $a_j$ is constant throughout, at minimum reflux

$$\phi_{s(j-1)} = \phi_{fj} = \phi_{rj};$$

when

$$(h+1) \leqslant j \leqslant l \tag{4}$$

These values of $j$ correspond to the $\phi_{sj}$'s, $\phi_{fj}$'s and $\phi_{rj}$'s having values between $a_h$ and $a_l$.

**Over-all Balance Equations.** Assume that the relative volatilities remain constant throughout the column. Furthermore, in the considerations to follow assume for simplicity that the product specifications are stated as the separation ratios of the keys.

Suppose, initially, that the keys are adjacent components. In order to determine the minimum reflux ratio, first calculate the quantity $\phi_{fl}$, where $\phi_{fl}$ is the one root fo $\Omega_f(\phi_{fj}) = 0$ lying between $a_h$ and $a_l$. But as was pointed out previously $\phi_{fl} = \phi_{rl}$ so that $\Omega_r(\phi_{fl}) = 0$. That is

$$V_r = \frac{D}{1-\rho} = \sum_{j=1}^{c} \frac{Dx_{Dj}}{1-\frac{\phi_{fl}}{a_j}} \tag{5}$$

Since at minimum reflux the components lighter and heavier than the keys go entirely into either the top or bottom product, the amounts and compositions of these products are completely known. Equation (5) therefore gives $1/1-\rho$, and from it the reflux ratio $\rho/1-\rho$. This is the method proposed by Underwood (*14*).

However, when the system contains any number of components between the keys, the problem is more difficult since neither $D$ nor the distribution of the components between the keys is known in advance. However, as shown in the derivations, a group of equations like Equation (5), containing as unknowns the above quantities and the (minimum) reflux ratio, may be set up and solved analytically. The expression so obtained for $\rho$ is

$$\rho = v \frac{\sum_{j=h,l,L} \frac{\omega_j}{a_j} Dx_{Dj}}{\sum_{j=h,l,L} \omega_j Dx_{Dj}} \tag{6}$$

where

$$v = \frac{\prod_{i=h+1}^{l} \phi_{fi}}{\prod_{i=h+1}^{l-1} a_i} \tag{7}$$

$$\omega_j = \frac{\prod_{i=h+1}^{l-1}\left(1-\frac{a_i}{a_j}\right)}{\prod_{i=h+1}^{l}\left(1-\frac{\phi_{fi}}{a_j}\right)} \tag{8}$$

Equation (6) is the over-all balance equation. When the keys are adjacent, Equation (6) reduces to the Underwood-type equation, Equation (5). To calculate the minimum reflux ratio by use of Equation (6), the values of $\phi_f$ lying between $a_h$ and $a_l$ are calculated, by trial, from $\Omega_f(\phi_f) = 0$. For a system containing $z$ split keys, there are $(z+1)$ values of $\phi_f$ lying between $a_h$ and $a_l$. Substituting these values of $\phi_f$ in Equation (6) gives $\rho$, from which the minimum reflux ratio, $\rho/1-\rho$, follows.

To illustrate the application of Equation (6) to a particular system, consider Example (1). In this example the heavy key is component number $4(k = 4)$, while the light key is component number $7(l = 7)$, and components 5 and 6 are split keys. There are three components 8, 9, 10 lighter than the light key. Values of $L$ are then 8, 9, 10. For this example

$$v = \frac{\phi_{r5}\phi_{r6}\phi_{r7}}{a_5 a_6} \tag{9}$$

and the first term (the term for which $j = h = 4$) of

$$\sum_{j=h,l,L} \frac{\omega_j}{a_j} Dx_{Dj}$$

is

$$\frac{\left(1-\frac{a_5}{a_4}\right)\left(1-\frac{a_6}{a_4}\right)\left(\frac{Dx_{D4}}{a_4}\right)}{\left(1-\frac{\phi_{r5}}{a_4}\right)\left(1-\frac{\phi_{r6}}{a_4}\right)\left(1-\frac{\phi_{r7}}{a_4}\right)} \tag{10}$$

The next term $j = l = 7$ is obtained by replacing the subscript 4 in Equation (10) by the subscript 7. The remaining terms $j = L = 8, 9, 10$ are obtained in the same manner. The terms of

$$\sum_{j=h,l,L} \omega_j Dx_{Dj}$$

are obtained by multiplying the corresponding terms of

$$\sum_{j=h,l,L} \frac{\omega_j}{a_j} Dx_{Dj}$$

by $a_j$.

The total moles in the top product is given by

$$D = \sum_{j=h,l,L} \omega_j Dx_{Dj} - v \sum_{j=h,l,L} \frac{\omega_j}{a_j} Dx_{Dj} \tag{11}$$

Since the quantities involved in this equation are the same as those which appear in Equation (6) little additional work is required to calculate the total moles in the top product.

The moles of a split key in the top product are given by

$$\frac{Dx_{DK}}{a_K} = -\frac{1}{\omega' z} \sum_{j=h,l,L} \frac{\omega' j}{a_j} Dx_{Dj};$$

$$K = h+1, h+2, \ldots, \text{or } l-1 \tag{12}$$

where

$$\alpha'_f = \frac{\prod_{i=h+1,\neq K}^{l-1}\left(1-\frac{\alpha_i}{\alpha_f}\right)}{\prod_{i=h+1}^{l}\left(1-\frac{\phi_{ri}}{\alpha_f}\right)} \qquad (13)$$

Equations corresponding to (6), (11), and (13) for $1/\sigma$, $B$, and $Bx_{BK}/\alpha_K$ have also been derived (*7*). The same results are arrived at formally by replacing $L$ in Equations (6), (11), and (13) by $H$, where $H$ refers to all components heavier than the heavy key, and by replacing all of the $D$'s by $B$'s. These equations are more convenient for calculational purposes when the number of $H$ components of the system is small compared to the number of $L$ components.

Underwood (*15*) recognized that the calculation of minimum reflux for systems containing split keys involved the solution of a set of simultaneous equations of the type of Equation (5). His solutions were obtained numerically and were somewhat simplified by the specification of sharp separations.

**Pinch Balance Equation.** In the past, it has been customary to calculate minimum reflux by consideration of the conditions in the pinches. For this reason the algebraic theory has been used to develop two equations which relate the pinch compositions for an arbitrary system having variable flow, variable volatilities, and any number of split keys. Although they are applicable to systems having any number of split keys, the general forms of these equations are complicated, and they are therefore recommended for use only when the keys are adjacent. For systems in which the keys are adjacent, these equations are

$$\frac{\left(1-\frac{\phi_{ri}}{\alpha_{pri}}\right)\left(1-\frac{\phi_{sh}}{\alpha_{psh}}\right)}{\left(1-\frac{\phi_{ri}}{\alpha_{prh}}\right)\left(1-\frac{\phi_{sh}}{\alpha_{psi}}\right)} = 1 \qquad (14)$$

$$\frac{x_{pri}x_{psh}}{x_{prh}x_{psi}} = \left[1-\frac{\left(\frac{\phi_{ri}}{\alpha_{prh}}-1\right)}{x_{prh}}\sum_{L}\frac{x_{prL}}{1-\frac{\phi_{ri}}{\alpha_{prL}}}\right]\left[1-\frac{\left(1-\frac{\phi_{sh}}{\alpha_{psi}}\right)}{x_{psi}}\sum_{H}\frac{x_{psH}}{\frac{\phi_{sh}}{\alpha_{psH}}-1}\right] \qquad (15)$$

Equations (14) and (15) are exactly equivalent and give precisely the same results. Equation (14) is more convenient to use and has been designated the pinch balance equation. The expression on the left-hand side of this equation has been designated the pinch factor, $pf'$. Then the pinch balance equation in terms of the pinch factor is $pf' = 1$. Determination of minimum reflux by Equation (14) is a trial process. Briefly stated it consists of assuming a reflux for the calculation of $\phi_{sh}$ and $\phi_{ri}$ from the equations $\Omega_s(\phi_{sh}) = 0$ and $\Omega_r(\phi_{ri}) = 0$. When the minimum reflux ratio is assumed, the values of $\phi_{sh}$ and $\phi_{ri}$ so obtained satisfy the pinch balance equation.

Equation (15) is the theoretical analogue of the Colburn correlation (*3*), which is

$$\frac{x_{pri}x_{psh}}{x_{prh}x_{psi}} = \left(1-\sum_{L}C_r x_{prL}\right)\left(1-\sum_{H}\frac{\alpha_{psH}}{\alpha_{psh}}C_s x_{psH}\right) \qquad (16)$$

where $C_r$ is an empirical function of

$$\left(\frac{\alpha_{pri}}{\alpha_{prh}}-1\right)\frac{\alpha_{pri}}{\alpha_{prL}}$$

and $C_s$ is another function of

$$\left(\frac{\alpha_{psi}}{\alpha_{psh}}-1\right)\frac{\alpha_{psH}}{\alpha_{psh}}$$

**Discussion of Pinch Balance Equation.** The pinch balance equation is actually exact only when the volatilities are constant throughout. Although this expression is not exact for systems having variable volatilities, the minimum reflux can be calculated from it with good accuracy. Considerations upon which this statement is based are given herewith.

In the derivations, a general expression was used valid for systems having variable flows, volatilities, and any number of split keys. The expression is

$$\frac{x_{pri}x_{psh}}{x_{prh}x_{psi}} = (pf')(1-\Delta_1)(1-\Delta_2) \qquad (17)$$

where $\Delta_1$ is a complicated function only of conditions in the rectifying pinch, and $\Delta_2$ is a similar function only of conditions in the stripping pinch. $pf'$ is a function of conditions in both pinches. When the equations given above (below Equation (*2*)) for $x_{prh}$, $x_{pri}$, $x_{psh}$, and $x_{psi}$ are substituted in the left-hand side of Equation (17), the resulting expression becomes an identity, for any reflux less than total; that is, the right-hand side exactly equals the left-hand side when all quantities appearing are evaluated at the given reflux.

To obtain information pertaining to the minimum reflux ratio from this expression, it is necessary to relate the pinches in some manner. This is readily done when the volatilities are constant. For in this case, as is shown in the derivations, $pf' = 1$ for systems having any number of split keys. When the keys are adjacent, the fact that $pf' = 1$ follows immediately from Equation (14), since for constant $\alpha$ systems $\phi_{sh} = \phi_{ri}$. When the volatilities are constant, Equation (17) can therefore be written

$$\frac{x_{pri}x_{psh}/x_{prh}x_{psi}}{(1-\Delta_1)(1-\Delta_2)} = 1 \qquad (18)$$

or

$$pf' = 1 \qquad (19)$$

For variable $\alpha$ systems, there is no theoretical treatment to give the precise value of $pf'$. However, the Colburn correlation has been found to be of good accuracy for variable $\alpha$ systems, which suggests that $pf'$ is generally close to unity at minimum reflux. Numerical data summarized in Table 1 show that $pf'$ is close but not exactly equal to unity when the $\alpha$'s are not constant. Thus if Jenny's and Brown and Holcomb's values for the minimum reflux ratios are accepted, the values of $pf'$ at minimum reflux are respectively 1.0574 and 1.0296. It is also indicated, however, that the numerical dependence of $pf'$ on the assumed reflux ratio is such that a small inaccuracy in $pf'$ results in a still smaller error in the minimum reflux ratio. Therefore, it is recommended that $pf'$ always be taken equal to unity. Then

TABLE 1.—VALUES OF TERMS APPEARING IN EQUATION (14)

Summary of Detailed Calculations for Systems with Variable Volatility Ratios

| Example No. | Source | Assumed Reflux Ratio, $\frac{R}{1-R}$ | Calculated Values $\frac{x_{pri}x_{psh}}{x_{prh}x_{psi}}$ | $pf'$ | $(1-\Delta_1)(1-\Delta_2)$ |
|---|---|---|---|---|---|
| 2 | Jenny (*8*) | 0.95 * | 0.8868 | 1.0574 | 0.8380 |
| | | 0.96 | 0.8498 | 1.0129 | 0.8390 |
| | | 0.963† | ..... | 1.0000 | ..... |
| 3 | Brown and Holcomb (*1*) | 1.55 | 0.9148 | 1.1636 | 0.7863 |
| | | 1.618** | 0.8171 | 1.0296 | 0.7936 |
| | | 1.628 | 0.8040 | 1.0121 | 0.7946 |
| | | 1.635† | ..... | 1.0000 | ..... |

*, ** Values obtained by stepwise method Jenny (*), and Brown and Holcomb (**).
† Values obtained by present authors by straight line extrapolation to $pf' = 1$.

TABLE 2.—SUMMARY OF EXAMPLES AND RESULTS

| Example No. | Source | Number of Components | Split Keys | $\alpha$ Values | Minimum Reflux Ratio: Stepwise | Over-all Balance | Pinch Balance | Colburn Correlation |
|---|---|---|---|---|---|---|---|---|
| 1 | Authors | 10 | 2 | Constant | .... | 2.678 | ..... | .... |
| 2 | Jenny (3) | 6 | 0 | Variable | 0.95* | 0.967 | 0.963 | 0.96 |
| 3 | Brown and Holcomb (1) | 7 | 0 | Variable | 1.618 | 1.640 | 1.635 | 1.66 |
| (Others not shown herein) | Murdoch (9) | 4 | 0 | Constant | .... | 0.9373 | 0.9373 | .... |
| | Colburn (3) Table 10† | 4 | 1 | Constant | 2.09 | 2.097 | 2.097 | 2.10 |
| | Colburn (3) Table 11† | 4 | 1 | Constant | 1.24 | 1.238 | 1.238 | 1.239 |

* By interpolation between 0.90 and 1.00.
† Colburn's table number.

when the keys are adjacent, the minimum value of the reflux is found by trial, such that the corresponding $\phi$'s, when substituted in Equation (14), make $pf' = 1$. Details of the calculation are shown here by illustrative examples.

**Over-all Balance Equations for Variable $\alpha$.** The pinch balance equation $pf' = 1$ amounts to fixing the relationship between the $\phi_{rj}$'s and $\phi_{sj}$'s having values between the $\alpha$'s of the keys. Since this is the same sort of relation as is involved in the over-all balance equation, it appears plausible that this equation can, with the proper assignment of $\alpha$ values, give results of the same order of accuracy as does the pinch balance equation. Suppose that the conditions at minimum reflux for a variable $\alpha$ separation are worked out correctly, in some manner. Then $(1-\rho)$, each $y_{prj}$ and each $x_{Dj}$ are also correct (regardless of the number of split keys), and the same is true of the $\phi_{rj}$'s, when these are calculated by Equation (1), with $\Omega_r(\phi_r) = 0$. But the rectifying pinch composition equations show that the same relationship between $y_{prj}$ and $x_{Dj}$ would exist if the $\alpha$'s above the rectifying pinch were altered. In other words the conditions in and below the rectifying pinch are independent of the $\alpha$ values above that pinch.

An analogous statement is true for the stripping section; therefore at minimum reflux only the $\alpha$ values in and between the pinches are significant. Suppose, next, that it is possible to represent the fractionation adequately by assigning constant, mean values to the $\alpha$'s for the region between the pinches. The outside light ($L$) components are of major importance in the rectifying pinch. This, coupled with the nature of Equation (15), indicates that $\alpha_{prL}$ should be used. Similarly, $\alpha_{psH}$ is recommended for any outside heavy component ($H$). Since the distributed components appear in both pinches, an average of $\alpha_{pr}$ and $\alpha_{ps}$ is suggested. That is,

$$\alpha_H = \alpha_{psH},\ \alpha_k = \frac{\alpha_{prk} + \alpha_{psk}}{2},\ \alpha_L = \alpha_{prL} \qquad (20)$$

where $H = 1$ to $h-1$, $k = h$ to $l$, $L = l+1$ to $c$. If these $\alpha$ values are correct for the region in and between the pinches, they can equally well be used over the entire column for present purposes. The variable $\alpha$ examples given below indicate that on this basis the over-all balance equation gives numerical results close to those from the pinch balance equation, $pf' = 1$; the accuracy of either method is good. If for each distributed component ($k$), $\alpha_{prk}$ is actually equal to $\alpha_{psk}$, both methods give, of course, the same value for the minimum reflux ratio.

## Calculational Methods and Illustrative Examples

The minimum reflux was calculated for each of several systems by the over-all and pinch balance methods; results are summarized and compared with values given by other methods, in Table 2.

**Calculational Methods, Constant $\alpha$.** For computational purposes the formulas may for convenience be cleared partially of fractions; for example, $1/(1-\phi/\alpha)$ may be written $\alpha/(\alpha-\phi)$.

The steps involved in using the over-all balance method when the $\alpha$'s are constant throughout the column are as follows:

1. Calculate, by trial, the $\phi_f$'s having values between $\alpha_h$ and $\alpha_l$ from $\Omega_f(\phi_{ff}) = 0$ (Equation (3))
2. Evaluate $v$ (Equation (7))
3. Evaluate $\sum_{j=h,l,L} \frac{\omega_j}{\alpha_j} Dx_{Dj}$ (see Eq. (8))
4. Multiply each term in Item 3 by $\alpha_l$, giving $\sum_{j=h,l,L} \omega_j Dx_{Dj}$
5. Substitute Items 3 and 4 in Equation (6), giving $\rho$. The minimum reflux ratio equals $\rho/1-\rho$

The pinch balance method, being more laborious, is not recommended for use when the volatility ratios are constant.

**Calculational Methods, Variable $\alpha$.** Both the over-all and pinch balance methods require knowledge of the $\alpha$'s in the pinches; when the $\alpha$ variation in the column is considerable, the proper values must be determined by trial. Split keys, if present, affect the minimum reflux calculations indirectly, in that they modify the pinch temperatures and compositions, and therefore the $\alpha$ values. If there are split keys the over-all balance method is recommended since Equation (12) provides a check on the separation ratios of such components. When the keys are adjacent and the volatility ratios vary considerably, either method may be used; there is little difference in the labor involved.

Assume first that the keys are adjacent. Then for either method determine the $\alpha$'s in the pinch as follows:

1. Assume a value for the minimum reflux ratio.
2. Using the assumed reflux ratio calculate, by trial, the temperatures of the rectifying and stripping pinches, by pinch composition equations.
3. Evaluate the $\alpha$'s at these temperatures.

Using the volatility ratio corresponding to the assumed reflux ratio, the following procedure is suggested for determination of the minimum reflux by the pinch balance equation, $pf' = 1$.

1. Calculate $\phi_{rl}$ and $\phi_{rh}$ corresponding to the assumed reflux ratio from $\Omega_r(\phi_{rl}) = 0$ and $\Omega_r(\phi_{rh}) = 0$, respectively (Equations (1) and (2)).
2. Evaluate $pf'$ (Equation (14)). If $pf' = 1$, the assumed reflux ratio is the minimum reflux ratio.
3. If the value of $pf'$ obtained in Item 2 is not equal to unity, assume a second reflux ratio. For this reflux ratio, evaluate the corresponding $\alpha$'s, $\phi$'s, and $pf'$. If the volatility ratios corresponding to the first assumed reflux are considered reliable, then it is necessary to evaluate only the $\phi$'s and $pf'$ corresponding to the second reflux ratio. If the values of $pf'$ are reasonably close to

TABLE 3.—EXAMPLE (1)

| Component No. | Total Mole Fraction in Feed $X_f$ | Relative Volatility $\alpha$ | Separation Ratio $(sr)$ | $D_{XD}$ | $B_{XB}$ |
|---|---|---|---|---|---|
| 1 | 0.05 | 0.40 | ... | .... | 0.05 |
| 2 | 0.12 | 0.70 | ... | .... | 0.12 |
| 3 | 0.05 | 0.90 | ... | .... | 0.05 |
| 4 | 0.13 | 1.00 | 0.2 | 0.021667 | 0.108333 |
| 5 | 0.14 | 1.15 | ... | .... | .... |
| 6 | 0.09 | 1.25 | ... | .... | .... |
| 7 | 0.16 | 1.35 | 7.0 | 0.14 | 0.02 |
| 8 | 0.14 | 1.50 | ... | 0.14 | .... |
| 9 | 0.08 | 2.00 | ... | 0.08 | .... |
| 10 | 0.05 | 3.00 | ... | 0.05 | .... |

$1 = q = 0.4$

TABLE 4.—DETERMINATION OF MINIMUM REFLUX RATIO FOR EXAMPLE (1)

(1) $\phi_f$'s calculated from $\Omega_f(\phi_f) = 0$

$\phi_{f3} = 1.0504$

$\phi_{f2} = 1.2016$

$\phi_{f1} = 1.2890$

(2) $v = 1.1318$

(3) $\sum_{j=h,l,L} \frac{\omega_j}{\alpha_j} D_{EDj} = 1.3102$

(4) $\sum_{j=h,l,L} \omega_j D_{EDj} = 2.0366$

(5) $\rho = \frac{v \sum_{j=h,l,L} \frac{\omega_j}{\alpha_j} D_{EDj}}{\sum_{j=h,l,L} \omega_j D_{EDj}} = 0.7281$

(6) $\frac{\rho}{1-\rho} = 2.6778$

(7) $D = \sum_{j=h,l,L} \omega_j D_{EDj} - v \sum_{j=h,l,L} \frac{\omega_j}{\alpha_j} D_{EDj}$

$= 0.5537$

(8) $D_{EDs} = -\frac{\alpha_s}{\omega_s'} \sum_{j=h,l,L} \omega_j' D_{EDj} = 0.0679$

(9) $D_{EDs} = -\frac{\alpha_s}{\omega_s'} \sum_{j=h,l,L} \omega_j' D_{EDj} = 0.0542$

(10) $\sum_{j=h}^{c} D_{EDj} = 0.5538,$

which compares well with value obtained in Item (7)

unity, straight line interpolation can be used to determine the minimum reflux ratio.

For the over-all balance equation, the volatility ratios suggested for use are specified by Equation (20). Using these $\alpha$ values, the remainder of the procedure is the same as that outlined previously for constant volatility ratios. If the reflux ratio differs greatly from that assumed to evaluate the $\alpha_{pr}$'s and $\alpha_{ps}$'s, then it is recommended that the pinch $\alpha$'s be evaluated again using the reflux ratio calculated. Comparison of these two sets of pinch $\alpha$ values will indicate the reliability of the calculated reflux ratio. If the $\alpha$'s vary considerably with temperature, it may be necessary to repeat the above procedure using another set of $\alpha$ values.

TABLE 5.—EXAMPLE (2)

| Component | Component No. | Total Mole Fraction in Feed $X_f$ | Mole Fraction in Top Product $x_D$ | Mole Fraction in Bottom Product $x_B$ | Rectifying Pinch $\alpha_{pr}$ at 145° F. | Stripping Pinch $\alpha_{ps}$ at 235° F. | $\alpha$ Values for Over-all Balance Equation |
|---|---|---|---|---|---|---|---|
| $nC_3$ | 1 | 0.12 | ..... | 0.299 | .... | 0.27 | 0.27 |
| $nC_3$ | 2 | 0.11 | ..... | 0.274 | .... | 0.50 | 0.50 |
| $nC_4$ | 3 | 0.17 | 0.005 | 0.417 | 1.00 | 1.00 | 1.00 |
| $C_3$ | 4 | 0.25 | 0.411 | 0.010 | 2.17 | 1.84 | 2.01 |
| $C_2$ | 5 | 0.09 | 0.150 | ..... | 5.65 | .... | 5.65 |
| $C_1$ | 6 | 0.26 | 0.434 | ..... | 24.60 | .... | 24.60 |

Feed Conditions: $V_F - V_S = 1 - q = 0.666$
Tower Pressure: 300 lbs./sq.in.abs.
Key components; $(\alpha r)_h = 61.50$, $(\alpha r)_l = 0.01796$

TABLE 6.—EXAMPLE (3)

| Component | Component No. | Total Mole Fraction in Feed $X_f$ | Mole Fraction in Top Product $x_D$ | Mole Fraction in Bottom Product $x_B$ | Rectifying Pinch $\alpha_{pr}$ at 160° F. | Stripping Pinch $\alpha_{ps}$ at 226.5° F. | $\alpha$ Values for Over-all Balance Equation |
|---|---|---|---|---|---|---|---|
| $C_6H_{14}$ | 1 | 0.1716 | ..... | 0.2900 | ..... | 0.1022 | 0.1022 |
| $nC_5H_{12}$ | 2 | 0.1208 | ..... | 0.2040 | ..... | 0.487 | 0.487 |
| $iC_5H_{12}$ | 3 | 0.0732 | ..... | 0.1236 | ..... | 0.589 | 0.589 |
| $nC_4H_{10}$ | 4 | 0.1935 | 0.0097 | 0.3201 | 1.000 | 1.000 | 1.000 |
| $iC_4H_{10}$ | 5 | 0.0805 | 0.1073 | 0.0623 | 1.270 | 1.284 | 1.252 |
| $C_3H_8$ | 6 | 0.2714 | 0.6650 | ..... | 2.556 | ..... | 2.556 |
| $C_2H_6$ | 7 | 0.0890 | 0.2180 | ..... | 6.833 | ..... | 6.833 |

Feed Condition: Boiling Point Liquid.
Tower Pressure: 215 lbs./sq.in.abs.
Key components; $(\alpha r)_h = 0.021108$, $(\alpha r)_l = 1.1875$

TABLE 7.—DETERMINATION OF MINIMUM REFLUX RATIO FOR EXAMPLES (2) AND (3); OVER-ALL BALANCE METHOD

(Relative volatilities used are those stated in column 8 of Tables 5 and 6.)

Example (2)

(1) $\phi_f$'s calculated from $\Omega_f(\phi_f) = 0$, Equation (3)

$\phi_{f4} = 1.3850$

(2) $\sum_{L} \frac{x_{Dj}}{1 - \frac{\phi_{f4}}{\alpha_{pr5}}} + \sum_{j=h,l} \frac{x_{Dj}}{1 - \frac{\phi_{f4}}{\alpha_j}}$

$= \frac{1}{1-\rho} = 1.971$

(3) $\rho/1 - \rho = 0.967$

Example (3)

(1) $\phi_f$'s calculated from $\Omega_f(\phi_f) = 0$, Equation (3)

$\phi_{f3} = 1.1436$

(2) $\sum_{L} \frac{x_{Dj}}{1 - \frac{\phi_{f3}}{\alpha_{pr5}}} + \sum_{j=h,l} \frac{x_{Dj}}{1 - \frac{\phi_{f3}}{\alpha_j}}$

$= \frac{1}{1-\rho} = 2.640$

(3) $\rho/1 - \rho = 1.640$

TABLE 8.—DETERMINATION OF MINIMUM REFLUX RATIO FOR EXAMPLE (2) PINCH BALANCE METHOD

(Relative volatilities used are those stated in columns 7 and 8 of Table 4.)

Assumed $\frac{\rho}{1-\rho} = 0.95$

(1) $\phi_{r4}$ calculated from $\Omega_r(\phi_{r4}) = 0$, Equation (1) $\phi_{r4} = 1.4815$

(2) $\phi_{s3}$ calculated from $\Omega_s(\phi_{s3}) = 0$, Equation (2) $\phi_{s3} = 1.3913$

(3) $pf' = \frac{\left(1 - \frac{\phi_{r4}}{\alpha_{pr4}}\right)\left(1 - \frac{\phi_{s3}}{\alpha_{ps3}}\right)}{\left(1 - \frac{\phi_{r4}}{\alpha_{pr5}}\right)\left(1 - \frac{\phi_{s3}}{\alpha_{ps4}}\right)} = 1.0574$

Assumed $\frac{\rho}{1-\rho} = 0.96$

(1) $\phi_{r4}$ and $\phi_{s3}$ corresponding to $\frac{\rho}{1-\rho} = 0.96$ are calculated from $\Omega_r(\phi_{r4}) = 0$ and $\Omega_s(\phi_{s3}) = 0$, respectively.

$\phi_{r4} = 1.4865$

$\phi_{s3} = 1.3860$

(2) $pf' = \frac{\left(1 - \frac{\phi_{r4}}{\alpha_{pr4}}\right)\left(1 - \frac{\phi_{s3}}{\alpha_{ps3}}\right)}{\left(1 - \frac{\phi_{r4}}{\alpha_{pr5}}\right)\left(1 - \frac{\phi_{s3}}{\alpha_{ps4}}\right)} = 1.0129$

By straight line extrapolation to $pf' = 1$, the minimum reflux ratio obtained is 0.963.

**Illustrative Examples.** To illustrate the use of the over-all balance equation, the solution of an example in which the relative volatilities remain constant throughout the column is given in Table 4. Specifications are given in Table 3. The $\phi_f$'s having values between $\alpha_h$ and $\alpha_l$ are $\phi_{f3}$, $\phi_{f2}$, and $\phi_{f1}$. Upon calculation of these values, the minimum reflux ratio is readily obtained by carrying out the procedure as outlined above.

For Examples (2) and (3) in which the volatility ratios vary throughout the column, the minimum reflux ratio is determined by use of the over-all balance equation. Solutions are shown in Table 7. Statements of these examples are given in Tables 5 and 6.

To illustrate use of the pinch balance equation, the minimum reflux ratio for Example (2) is determined as shown in Table 8.

Since the minimum reflux 0.95 as given by Jenny was obtained by straight line interpolation between 0.90 and 1.0, it is subject to some error. For a reflux ratio of 0.95, the correct value of $pf'$ is 1.0574. But as pointed out previously inaccuracy in $pf'$ results in only a small error in the minimum reflux ratio. For $pf' = 1$, an error in $pf'$ of 5.74%, the minimum reflux ratio is only 1.37% greater than that obtained by Jenny.

## Conclusions

The over-all and pinch balance methods for calculation of minimum reflux are exact when the volatility ratios are constant throughout the column. When the volatility ratios vary within the column, neither method is exact, but either can still be used satisfactorily if appropriate mean values of the $\alpha$'s are employed. The method proposed for the determination of these $\alpha$ values is indicated by the examples to be adequate.

The over-all balance method is recommended for use when the volatility ratio variation within the column is small, and also when there are split keys even with more widely varying volatility ratios. When the volatility ratios vary considerably and the keys are adjacent, either method can be used; there is little difference in accuracy or in labor involved.


## Acknowledgment

This research was supported in part by The Texas Engineering Experiment Station; this aid is gratefully acknowledged.


## Notation

$B$ = total moles of bottom product per mole of feed

$D$ = total moles of top product per mole of feed

$pf'$ = a function of both rectifying and stripping section pinches

$q$ = heat required to bring one mole of feed from inlet to feed-plate temperature and vaporize it, divided by latent heat of vaporization of feed

$(K)$ = vapor-liquid molal equilibrium ratio: $y/x$;

$(K)_b = \dfrac{1}{\sum \alpha_i x_i} = \sum y_i/\alpha_i$ = vapor-liquid equilibrium ratio, $y/x$, of the base component (i.e., the component for which $\alpha = 1$)

$v$ = factor appearing in the expression for minimum reflux; defined by Equation (7)

$X_{fi}$ = mole fraction of $i$'th component in total feed

$x_{pri}$ = mole fraction of $i$'th component in liquid in the rectifying section pinch

$x_{psi}$ = mole fraction of $i$'th component in liquid in the stripping section pinch

$y_{pri}$ = mole fraction of $i$'th component in the vapor in rectifying section pinch

$y_{psi}$ = mole fraction of $i$'th component in vapor in stripping section pinch

*Subscripts:*

$b$ = standard component on which $(K)_b$ and $\alpha$ are based. May be chosen at will

$B$ = bottom product

$c$ = all components, total number of components, or component having largest $\alpha$ value (as indicated by context)

$D$ = top product

$f$ = column feed

$h$ = heavy or less volatile key component

$H$ = any component for which $\alpha < \alpha_h$

$i, j$ = component numbers (between 1 and $c$). Components are arranged in order of increasing volatility, 1 being the least and $c$ being the most volatile

$K$ = any component having a volatility ratio between those of the keys

$l$ = light or more volatile key component

$L$ = any component for which $\alpha > \alpha_l$

$pr$ = rectifying section pinch

$ps$ = stripping section pinch

$r$ = rectifying section

$s$ = stripping section

*Greek Letters:*

$\alpha$ = volatility ratio of individual components; $(K)_i/(K)_b$

$\Delta_1$ = function of rectifying pinch conditions

$\Delta_2$ = function of stripping section pinch conditions

$\prod$ = product of factors

$\rho = L_r/V_r$ (molal ratio of liquid to vapor flows) in rectifying section

$\sigma = V_s/L_s$ (molal ratio of vapor to liquid flows) in stripping section

$\sum$ = summation symbol

$\sum_c$ = sum over all components

$\phi_{fj}$ = $j$'th root of Equation (3); $(j = 1 \text{ to } c)$

$\phi_{rj}$ = $j$'th root of Equation (1); $(j = 1 \text{ to } c)$, and $\phi_{rh} = \rho/K_{pr,b}$

$\phi_{sj}$ = $j$'th root of Equation (2); $(j = 1 \text{ to } c)$, and $\phi_{sl} = 1/(\sigma K_{ps,b})$

$\Omega_f(\phi)$ = function defined by Equation (3)

$\Omega_r(\phi)$ = rectifying function; defined by Equation (1)

$\Omega_s(\phi)$ = stripping section function; defined by Equation (2)

$\omega_j$ = ratio of factors; defined by Equation (8)

$\omega_j'$ = ratio of factors; defined by Equation (13)

*Mathematical Symbols:*

$\equiv$ means identically equal to

$\leqslant$ means equal to or less than

$\geqslant$ means equal to or greater than

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# THERMODYNAMICS OF THE DEACON PROCESS

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THE equilibrium reaction involving hydrogen chloride, oxygen, chlorine, and water,

$$4HCl + O_2 \rightleftharpoons 2Cl_2 + 2H_2O \quad (1)$$

provides an interesting example of the feasibility of altering process variables in order to secure a product for which there is an economic demand. This reaction has been of commercial interest since 1868 when H. W. Deacon first successfully performed the oxidation of hydrogen chloride with air on a continuous basis. At that time the large quantities of by-product hydrogen chloride arising from the Leblanc soda-ash process yielded a low-cost raw material for the production of the more valuable chlorine. The Deacon process enjoyed considerable success for a number of years; and then, due both to the passing of the Leblanc process and to distinct disadvantages, involved in the method, gave way almost entirely to electrolytic processes.

During the early twenties caustic soda was the main product of the electrolytic cells, while chlorine was considered a by-product. In order to satisfy the demand for caustic, the level of production was so great as to cause considerable oversupply of chlorine. This cheap and abundant supply of chlorine led to its use as a raw material in the production of hydrogen chloride. By a slight change of process variables, namely, increasing the temperature, equilibrium in reaction 1 was shifted to the left to the extent that the reaction between chlorine and steam was used as a commercial source of hydrogen chloride (*26*).

During the past few years the Deacon process has attracted increasing attention as a means of both producing chlorine and making use of tremendous quantities of by-product hydrogen chloride arising in the organic-synthesis industries. The availability of cheap tonnage oxygen, the discovery of newer and better methods of catalysis, and improvements in general plant layout have eliminated many of the objections to the process.

As the basic chemical reaction of the Deacon process is one of equilibrium, data for the effect of various variables on this equilibrium are highly desirable. The purpose of this paper is twofold: (1) to calculate from spectroscopic data the thermodynamic functions of hydrogen chloride and chlorine and (*2*), using these values together with the latest functions for water vapor and oxygen, to calculate the equilibrium conditions for reaction 1. In particular, the effects of temperature, pressure, impurities, and ratio of reactants will be studied.

## Recent Developments

Although the oxidation of hydrogen chloride with air had been attempted as early as 1845, it was not until 1868 that Deacon (*5*) made the process successful by arranging the reactions to form a continuous operation. The process enjoyed considerable success for years and served to support the Leblanc process even after the ammonia-soda process began to undersell the Leblanc soda and chemical caustic. However, the development of the electrolytic caustic-chlorine cell eliminated the Deacon process as a source of commercial chlorine.

In recent years the Deacon process has attracted renewed attention because of two economic factors. The insatiable demand for chlorine has forced an examination of all processes for its production, particularly those that do not produce caustic (*23*). The hydrogen chloride, or hydrochloric acid, produced in the chlorination of organic compounds gives a by-product that must be sold or utilized. Because 50% of the chlorine used in chlorination reactions becomes hydrogen chloride, the reformation of chlorine is frequently desired.

Many of the objectionable features of the Deacon process have been overcome. The availability of industrial oxygen at low cost has largely eliminated the inert nitrogen, so that now a high concentration of chlorine can be obtained in the product gas. A great deal of work has been done on improvement of the catalyst and process procedure. Catalysts include compounds of copper, iron, manganese, and chromium, for which an impressive list of patents has been issued recently (*1-4, 6, 7, 13, 16-18, 25, 32, 33, 35-38*).

Two examples of recent developments in the Deacon process are a process developed by the Air Reduction Co. (*1, 2, 32*) and a German process that reached the pilot-plant stage during World War II (*23*). Both use industrial oxygen rather than air as the oxidizing agent. The former process uses the chlorides of rare-earth metals as promoters of catalyst activity and maintains the catalyst in the reaction zone by condensation of volatilized catalyst and reversal of gas flow through the catalyst chamber. The latter process uses a molten mixture of potassium chloride and ferric chloride as the catalyst. The former process uses compression and refrigeration to remove the chlorine; whereas the latter uses absorption in sulfur monochloride, thereby leaving the unused oxygen and hydrogen chloride to be recirculated. A cost estimate prepared by the Germans indicated that a plant producing 50 tons or more per day could produce chlorine at considerably less cost than by electrolysis of hydrochloric acid.

**Thermodynamic Functions.** To secure the most accurate data for the calculation of equilibrium conditions, the thermodynamic functions of chlorine and hydrogen chloride were calculated from the latest spectroscopic constants. The method used was essentially that of Mayer and Mayer (*31*). Although this is an approximation method, the results have been found to be in excellent agreement with those found by the approximation method of Gordon and Barnes (*15*) and the exact method of Giauque and Overstreet (*14*). The physical constants were the latest published by the National Bureau of Standards (*40*). Account was taken of the relative abundance of the two chlorine isotopes (*41*) although the hydrogen isotopes were not considered separately. The molecular constants for hydrogen chloride were those of Lindholm (*30*) for $HCl^{35}$, and these were converted to $HCl^{37}$ by the method of Herzberg (*19*). The values of the molecular constants for chlorine were those of Herzberg (*20*) and Elliott (*9*) for $Cl_2^{35\text{-}35}$, and these were calculated to $Cl^{35}$—$Cl^{37}$ and $Cl_2^{37}$ by the same method (*19*). For hydrogen chloride the values of the thermodynamic functions were calculated separately for the isotopes, and these values then combined to give average molal values. For chlorine, molal-average constants were used for the calculations, which are well within the limits of error of the method.

The calculated values are given in Tables 1 and 2. These values agree well with experimental values of entropy

TABLE 1

Thermodynamic Functions of HCl
Units: gram moles, calories, Kelvin degrees

| T°K | $-\frac{(G^\circ - H_0^\circ)}{T}$ | $\frac{(H^\circ - H_0^\circ)}{T}$ | $S^\circ$ | $C_p^\circ$ |
|---|---|---|---|---|
| 200 | 34.986 | 6.909 | 41.895 | 6.961 |
| 273.16 | 37.142 | 6.923 | 44.065 | 6.963 |
| 298.16 | 37.748 | 6.926 | 44.674 | 6.964 |
| 300 | 37.791 | 6.926 | 44.717 | 6.964 |
| 400 | 39.785 | 6.937 | 46.722 | 6.973 |
| 500 | 41.334 | 6.945 | 48.280 | 7.004 |
| 600 | 42.602 | 6.961 | 49.563 | 7.068 |
| 700 | 43.676 | 6.983 | 50.659 | 7.167 |
| 800 | 44.610 | 7.013 | 51.623 | 7.289 |
| 900 | 45.439 | 7.051 | 52.490 | 7.423 |
| 1000 | 46.184 | 7.095 | 53.279 | 7.560 |
| 1100 | 46.862 | 7.144 | 54.006 | 7.693 |
| 1200 | 47.486 | 7.195 | 54.681 | 7.819 |
| 1300 | 48.064 | 7.247 | 55.311 | 7.936 |
| 1400 | 48.603 | 7.300 | 55.903 | 8.043 |
| 1500 | 49.108 | 7.353 | 56.461 | 8.141 |
| 1600 | 49.585 | 7.405 | 56.990 | 8.230 |
| 1700 | 50.035 | 7.456 | 57.491 | 8.310 |
| 1800 | 50.463 | 7.506 | 57.969 | 8.382 |
| 1900 | 50.870 | 7.553 | 58.423 | 8.449 |
| 2000 | 51.258 | 7.600 | 58.858 | 8.509 |

TABLE 2

Thermodynamic Functions of $Cl_2$
Units: gram moles, calories, Kelvin degrees

| T°K | $-\frac{(G^\circ - H_0^\circ)}{T}$ | $\frac{(H^\circ - H_0^\circ)}{T}$ | $S^\circ$ | $C_p^\circ$ |
|---|---|---|---|---|
| 200 | 43.050 | 7.108 | 50.158 | 7.576 |
| 273.16 | 45.293 | 7.293 | 52.586 | 7.999 |
| 298.16 | 45.934 | 7.357 | 53.291 | 8.112 |
| 300 | 45.979 | 7.362 | 53.341 | 8.119 |
| 400 | 48.130 | 7.594 | 55.724 | 8.438 |
| 500 | 49.846 | 7.783 | 57.629 | 8.624 |
| 600 | 51.279 | 7.934 | 59.213 | 8.742 |
| 700 | 52.511 | 8.056 | 60.567 | 8.822 |
| 800 | 53.594 | 8.155 | 61.749 | 8.880 |
| 900 | 54.559 | 8.238 | 62.797 | 8.924 |
| 1000 | 55.431 | 8.309 | 63.740 | 8.960 |
| 1100 | 56.225 | 8.369 | 64.594 | 8.990 |
| 1200 | 56.956 | 8.422 | 65.378 | 9.015 |
| 1300 | 57.632 | 8.468 | 66.100 | 9.038 |
| 1400 | 58.261 | 8.510 | 66.771 | 9.059 |
| 1500 | 58.850 | 8.547 | 67.397 | 9.078 |
| 1600 | 59.402 | 8.581 | 67.983 | 9.096 |
| 1700 | 59.923 | 8.612 | 68.535 | 9.112 |
| 1800 | 60.416 | 8.640 | 69.056 | 9.128 |
| 1900 | 60.884 | 8.666 | 69.550 | 9.144 |
| 2000 | 61.329 | 8.690 | 70.019 | 9.159 |

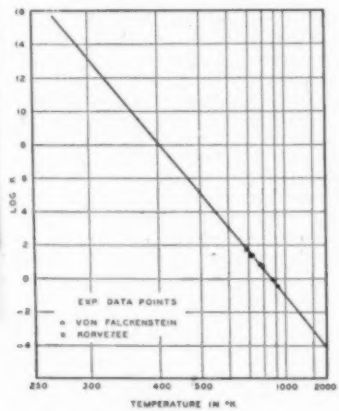


Fig. 1. Equilibrium constant of Deacon process as a function of temperature.

(*14*) and heat capacities (*34*). The values published by the National Bureau of Standards (*39*) are in close agreement with these values. Their calculations were based on the molecular constants of Spencer and Justice (*42*), which in turn were derived from the work of Elliott (*8*) and Kemble (*24*), whose work predates that used in this paper.

Algebraic equations were derived to fit the heat-capacity values as nearly as possible. In order to make the equations of equal reliability over the entire range covered, an extension of the method of least squares (*27*) was used to give a minimum percentage error rather than a minimum absolute error. Empirical constants for a cubic equation are given in Table 3. The values over the temperature range of 273° to 1500° K. are those reported by Kobe and Long (*28*).

The values of the thermodynamic functions for oxygen and water vapor which have recently been recalculated by the National Bureau of Standards (*39, 43*) were used. The Bureau values for heats of formation of water and hydrogen chloride were used also.

**Equilibrium Constants.** The equilibrium constant of the reaction was calculated from the following relationship:

$$-\frac{\Delta G^\circ}{T} = 2.3026\ R \log K \qquad (2)$$

The values are given in Table 4. A graph (Figure 1) of log $K$ with respect to the reciprocal of the absolute temperature showed an almost linear relationship. The equation of the best straight line through the points was determined to be

$$\log K = \frac{6{,}104.4}{T} - 7.0994 \qquad (3)$$

The maximum deviation using Equation (3) is 0.08 in the value of log $K$, and the average deviation is 0.032. A more accurate relationship is given by Equation (4), which has a maximum deviation of 0.005 and an average deviation of 0.0007.

$$\log K = \frac{5881.7}{T} - 0.930,35 \log T + (1.3704 \times 10^{-4})T - (1.7581 \times 10^{-8})T^2 - 4.1744 \qquad (4)$$

The equilibrium constants calculated from these spectroscopic data compare favorably with accepted experimental values. The experimental values of Von Falckenstein (*10-12*) and Korvezee (*29*) are plotted on Figure 1 with the calculated curve.

TABLE 3.—EMPIRICAL CONSTANTS FOR HEAT-CAPACITY EQUATIONS
$C_p^\circ = a + bT + cT^2 + dT^3$

| Gas | Temp. Range °K. | $a$ | $b \cdot 10^3$ | $c \cdot 10^6$ | $d \cdot 10^9$ | Average deviation % | Maximum deviation % |
|---|---|---|---|---|---|---|---|
| HCl ..... | 200–2000 | 7.053 | −0.8712 | 1.916 | − 0.5635 | 0.25 | 0.50 |
| HCl ..... | 273–1500 | 7.244 | −1.820 | 3.170 | −103.6 | 0.08 | 0.22 |
| $Cl_2$ ..... | 200–2000 | 6.860 | 4.994 | −3.745 | 0.9214 | 0.65 | 1.85 |
| $Cl_2$ ..... | 273–1500 | 6.8214 | 5.7095 | −3.107 | 154.7 | 0.23 | 0.50 |

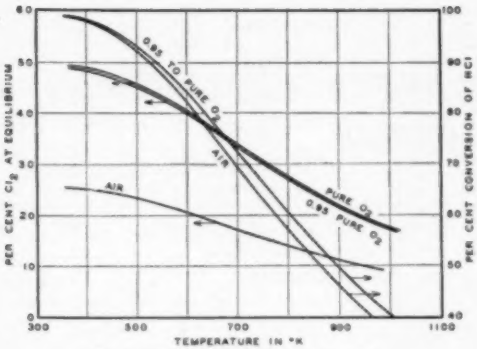


Fig. 2. Effect of purity of oxidizing agent.

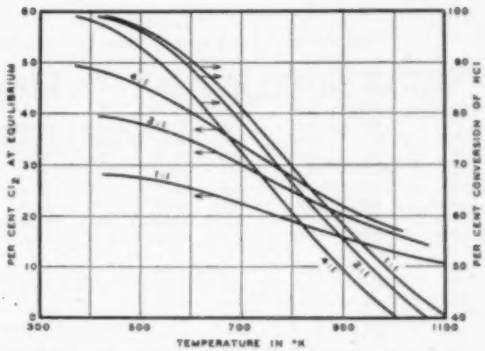


Fig. 3. Effect of ratio of reactants. HCl : $O_2$ ratio indicated on curve.

## Effect of Process Variables

Calculations were made to show the effect of temperature, pressure, inert gases and the ratio of reactants on the equilibrium conversion of hydrogen chloride to chlorine. The equilibrium constant can be expressed in terms of the activities of the components of Equation (1):

$$K = \frac{(a_{H_2O})^2(a_{Cl_2})^2}{(a_{HCl})^4(a_{O_2})} \qquad (5)$$

All components are in the gaseous state, and so the standard state is the component gas at unit fugacity expressed in atmospheres. Using the nomenclature of Hougen and Watson (*22*), the following equations can be written for the calculation of activity:

$$a_B = f_B = N_B f_{Bv} = N_B p_{Bv} \pi \qquad (6)$$

The mole fraction, $N_B$, can be calculated from the total moles present:

$$N_B = \frac{n_B}{n_{HCl} + n_{O_2} + n_{H_2O} + n_{Cl_2} + n_{inert}} \qquad (7)$$

A term $K_v$ can be calculated from the fugacity coefficients:

$$K_p = \frac{(p_{H_2O})^2(p_{Cl_2})^2}{(p_{HCl})^4(p_{O_2})} \qquad (8)$$

Values of the fugacity coefficients were read from the generalized chart of Hougen and Watson (*21*). At atmospheric pressure the values for all gases are close to unity and so $K_p$ has a value of unity. At higher pressure values were calculated using a trial-and-error method to secure the necessary value of temperature. Equation (9) represents the equation from which all equilibrium calculations were made.

$$K = \frac{(n_{H_2O})^2(n_{Cl_2})^2}{(n_{HCl})^4(n_{O_2})} \cdot K_p \cdot \frac{n_{HCl} + n_{O_2} + n_{H_2O} + n_{Cl_2} + n_{inert}}{\pi} \qquad (9)$$

Calculations were made by assuming a degree of conversion, calculating the corresponding equilibrium constant, and then reading the temperature from Figure 1 on an enlarged scale. The results are given in Table 5 and shown in Figures 2, 3, and 4.

All the equilibrium calculations were made on the assumption that all four reacting materials were in the gaseous state. At low temperatures or at high pressures, conditions might lead to the condensation of water within the reaction zone. If the condensed water could be assumed to be pure, its activity would be taken as unity, a value which would lead to considerable change in the calculated equilibrium percentages of chlorine. The actual calculations in such a case would be rather difficult involving a great deal of trial-and-error work. However, a further complication would

TABLE 4
Thermodynamic Data and Equilibrium Constants for Deacon Reaction

| T°K | $-\Delta S°$ (cal/deg) | $-\Delta H°$ (cal) | $-\Delta G°$ (cal/deg.) | log K |
|---|---|---|---|---|
| 298.16 | 30.905 | 27,345 | 60.809 | 13.290 |
| 300 | 30.926 | 27,351 | 60.243 | 13.166 |
| 400 | 31.553 | 27,566 | 37.363 | 8.1656 |
| 500 | 31.897 | 27,720 | 23.543 | 5.1453 |
| 600 | 32.120 | 27,838 | 14.277 | 3.1202 |
| 700 | 32.260 | 27,926 | 7.635 | 1.6686 |
| 800 | 32.378 | 28,008 | 2.632 | 0.5752 |
| 900 | 32.490 | 28,094 | -1.275 | -0.2786 |
| 1000 | 32.594 | 28,181 | -4.413 | -0.9645 |
| 1100 | 32.701 | 28,283 | -6.989 | -1.5274 |
| 1200 | 32.796 | 28,386 | -9.141 | -1.9977 |
| 1300 | 32.889 | 28,488 | -10.975 | -2.3986 |
| 1400 | 32.975 | 28,589 | -12.554 | -2.7437 |
| 1500 | 33.049 | 28,695 | -13.919 | -3.0420 |
| 1600 | 33.119 | 28,792 | -15.124 | -3.3053 |
| 1700 | 33.178 | 28,883 | -16.186 | -3.5379 |
| 1800 | 33.238 | 28,975 | -17.141 | -3.7461 |
| 1900 | 33.284 | 29,051 | -17.994 | -3.9326 |
| 2000 | 33.333 | 29,136 | -18.765 | -4.1011 |

Fig. 4. Effect of pressure. ▼

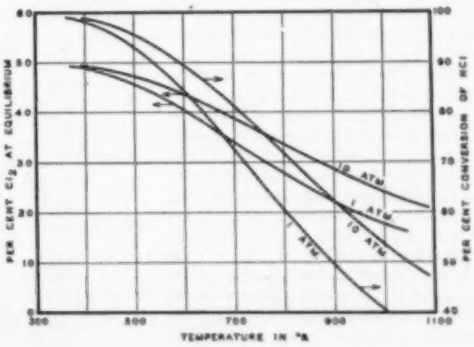

TABLE 5

Effect of Process Variable on the Equilibrium in the Deacon Process

| Conversion of HCl % | Case I Temp. °K | Case I Equil. $Cl_2$ Content,% | Case II Temp. °K | Case II Equil. $Cl_2$ Content,% | Case III Temp. °K | Case III Equil. $Cl_2$ Content,% | Case IV Temp. °K | Case IV Equil. $Cl_2$ Content,% |
|---|---|---|---|---|---|---|---|---|
| 99 | 370 | 49.38 | 364 | 25.46 | 368 | 48.74 | 419 | 39.52 |
| 97.5 | 426 | 48.45 | 418 | 25.04 | 426 | 47.82 | 477 | 38.80 |
| 95 | 473 | 46.91 | 463 | 24.32 | 473 | 46.31 | 524 | 37.62 |
| 90 | 541 | 43.90 | 528 | 22.89 | 540 | 43.35 | 588 | 35.29 |
| 80 | 636 | 38.10 | 619 | 20.10 | 636 | 37.62 | 686 | 30.77 |
| 70 | 722 | 32.56 | 699 | 17.37 | 721 | 32.16 | 772 | 26.42 |
| 60 | 805 | 27.27 | 776 | 14.70 | 805 | 26.94 | 853 | 22.22 |
| 50 | 894 | 22.22 | 862 | 12.10 | 893 | 21.96 | 951 | 18.18 |
| 40 | 1005 | 17.39 | 966 | 9.57 | 1004 | 17.19 | 1062 | 14.29 |

| Conversion of HCl % | Case V Temp. °K | Case V Equil. $Cl_2$ Content,% | Case VI Temp. °K | Case VI Equil. $Cl_2$ Content,% |
|---|---|---|---|---|
| 99 | 429 | 28.25 | 396 | 49.38 |
| 97.5 | 490 | 27.36 | 460 | 48.45 |
| 95 | 539 | 26.95 | 515 | 46.91 |
| 90 | 608 | 25.35 | 595 | 43.90 |
| 80 | 709 | 22.22 | 714 | 38.10 |
| 70 | 797 | 19.18 | 820 | 32.56 |
| 60 | 885 | 15.22 | 924 | 27.27 |
| 50 | 980 | 13.33 | 1047 | 22.22 |
| 40 | 1108 | 10.53 | | |

Case I. Stoichiometric ratio of reactants, pure oxygen as oxidising agent, and one atmosphere pressure

Case II. Stoichiometric ratio of reactants, air as oxidising agent, and one atmosphere pressure

Case III. Stoichiometric ratio of reactants, 95% pure oxygen as oxidising agent, and one atmosphere pressure

Case IV. 4:2 ratio of HCl to $O_2$, pure oxygen as oxidising agent, and one atmosphere pressure

Case V. 4:4 ratio of HCl to $O_2$, pure oxygen as oxidising agent, and one atmosphere pressure

Case VI. Stoichiometric ratio of reactants, pure oxygen as oxidising agent, and 10 atmospheres pressure

arise because both HCl and $Cl_2$ are exceedingly soluble in water, particularly under the higher pressures or lower temperatures that would lead to condensation. This fact would not only lead to a change in the activity of the water but to a change in the availability of these two materials for the reaction.

Under present plant-operation conditions the possibility of condensation of water vapor seems rather remote. For the case of stoichiometric ratio of reactants, 1 atm. pressure, and pure oxygen as the oxidizing agent, condensation will begin at about 80° C. For similar conditions but with 10 atm. pressure, water begins to condense at about 150° C. With present catalysts, the rate of reaction drops so rapidly with decreasing temperature that the process is not commercially feasible below about 430° C. (*26*). Most commercial processes are carried out at a temperature of about 450° C. to establish a balance between low conversion rates at low temperatures and excessive volatilization of the catalyst at higher temperatures. Even considering the possibility of the discovery of newer catalysts giving rapid conversion rates at lower temperatures, a high degree of conversion is reached well above the condensation point of water.

It is seen that no essential difference in conversion exists between pure oxygen and a 95% pure industrial oxygen, though the concentration of chlorine in the equilibrium mixture is slightly less. Although increasing the excess oxygen from 0 to 300% will increase the conversion by about 10% at 700° K., the chlorine content of the resulting gas is decreased by about 12%. The increased conversion must be balanced against the increased cost of recovery of the chlorine from the more dilute gas. Increased pressure will give both increased conversion and increased chlorine content.

## Summary

New calculations have been made for the thermodynamic properties of chlorine and hydrogen chloride. These data have been combined with those of the National Bureau of Standards to calculate equilibrium conversion and concentration of chlorine for the Deacon process:

$$4HCl + O_2 \rightleftharpoons 2Cl_2 + 2H_2O \quad (1)$$

The use of industrial oxygen rather than air gives a small increase in conversion and a greatly increased concentration of chlorine in the equilibrium gas. Rate of reaction should also be increased. Economic-balance calculations can be based on the results presented here.

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## CORRECTION

In "An Empirical Equation for Thermodynamic Properties of Light Hydrocarbons and Their Mixtures," by Messrs. Benedict, Webb and Rubin, Vol. 47, page 421 (1951), Table 1, constant "a" should be 0.0494000 instead of 0.494000.

# CONTINUOUS EXTRACTION IN A MULTISTAGE MIXER COLUMN

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**Performance data are presented for a continuous countercurrent extraction column, composed of a 6-in. diam. vertical glass tube, compartmented by horizontal plates, agitated by mixing impellers on a vertical shaft, and baffled by vertical members. Acetic acid was extracted from water to methyl-isobutyl ketone, and also from the ketone to water, in a countercurrent manner using the column with different-sized compartments, impellers, impeller positions, impeller speeds, acid concentrations, and feed rates. Settling zones were provided above and below the mixing-extracting compartments, and no coalescence was allowed between compartments.**

**Throughputs as high as 4400 lb./(sq.ft.)(hr.) were investigated (combined streams and based on column cross-section area). The best stage efficiency showed a minimum height for a theoretical contact stage of 3.7 in. for a throughput of 2140 lb./(sq.ft.)(hr.) using eight compartments, each 3 in. high. This corresponds to a stage efficiency of 81%. Comparisons are made with the performance of other types of extraction columns.**

**Batch contacting was used as a guide to the column operation.**

MOST countercurrent multistage extraction processes have made use of a series of mixers and settlers. In these systems two relatively immiscible phases are brought into intimate contact by mixing in one vessel and then allowed to separate in another nonagitated vessel; this combination constitutes a stage. The two phases pass countercurrently from stage to stage through the system, hence the name multistage countercurrent operation. Mixer-settler arrangements with individual mixing and settling tanks and connecting piping and pumps are complicated and in some cases are large and bulky, but because of their capacity and simple mechanism they enjoy considerable use.

Many continuous extraction columns have been described in the technical and patent literature (*4, 9, 14, 17*). Vertical cylindrical columns have been employed to contact two immiscible liquids by placing in them spray nozzles, perforated plates, bubble-cap plates, packing, rotating impellers, pulsating flows, and various compartmentations and combinations of these items. Extraction efficiencies for most commercial extraction towers which involve more than one equilibrium contact are relatively low. Multiple mixers and settlers have, therefore, been used often for large-scale extraction equipment. Other means of separation, such as distillation or evaporation, have been used in preference to liquid extraction whenever possible because of the relatively poor performance of extraction columns.

Vertical cylindrical towers containing mechanical agitators or mixing impellers have been used for extraction for a number of years but comprehensive performance data necessary for process engineering design have not been available. Recently, data were published (*14, 15*) for a column consisting of alternate vertical packed and mixed sections. Such a column is similar in principle to the mixer-settler multistage unit.

Several commercial extraction columns were installed during the recent war, consisting of a vertical cylinder, compartmented by horizontal plates having openings through which a vertical shaft was placed and turbine-type mixing impellers placed on the shaft. Each compartment was baffled by vertical baffles and one impeller used for each compartment. Four vertical baffles were placed in each compartment to give the desired mixing flow pattern (*13*) and when the mixers were rotated, a dispersed phase was formed in each compartment. Only two settling sections were present, at the top and bottom of the column. The purpose of this type extraction unit was: to provide dispersion of drops of one phase in another by mixing impeller action; to provide forced turbulence to the continuous phase to lower its mass-transfer resistance; to produce countercurrent stagewise flow; and to eliminate the interstage settling spaces. It was desired to produce as nearly as possible an equilibrium contact between the dispersed phase flowing countercurrent by gravity and the continuous phase in each successive compartment. The dispersed phase was formed by the action of the fluid motion generated by the rotating impeller and boundaries. These columns were found to be operable in a countercurrent stagewise manner. Accordingly, a glass wall column of this type was constructed for the work here reported to obtain performance data for such units, and to evaluate some of the many variables involved in process and equipment design of this type equipment.

The column performance has been evaluated for the chemical system of methyl isobutyl ketone, water, and acetic acid. The extraction performance was determined as a function of the following variables:

1. Liquid flow rates. 2. Impeller speed. 3. Impeller diameter. 4. Impeller position. 5. Compartment height. 6. Diameter of opening between compartments. 7. Direction of solute diffusion and solute concentration. 8. Total number of compartments and stages. Dependent variables such as drop size, separation or coalescence rates of the drops, and flooding phenomena were also observed and evaluated.

## Preliminary Batch Experiments

One of the important conditions in experimental liquid-liquid extraction involving drops is that drop size can be reproduced and that they will settle or coalesce in the same way for repeat experiments. In other words, the chemical system must be stable and the physical and chemical properties which have an effect on drop size and coalescence must remain constant throughout the work. It is noteworthy that many systems will not remain stable for a necessary period of days or weeks. Considerable work

Tables 7-14 of this paper are on file (Document 3625) with American Documentation Institute, 1719 N Street, Northwest, Washington, D. C. Microfilm and photoprints obtainable by remitting $1.00 and $1.35, respectively. (See Table 3 for content of tables.)

* Present address: Mixing Equipment Co., Rochester, N. Y.

Table 1

Physical Properties and Equilibrium Data for the System Methyl Isobutyl Ketone, Water, and Acetic Acid

| | Water Layer | | | Ketone Layer | | |
|---|---|---|---|---|---|---|
| Temp. °F. | Weight % Acid | Specific Gravity | Viscosity Centipoises | Weight % Acid | Specific Gravity | Viscosity Centipoises |
| 64 | 1.29 | | | 0.790 | | |
| | 2.54 | | | 1.61 | | |
| 66 | 1.29 | | | 0.792 | | |
| | 2.54 | | | 1.61 | | |
| 73 | 1.302 | 0.990 | 1.13 | 0.796 | 0.803 | 0.68 |
| | 2.54 | 1.00 | 1.17 | 1.60 | 0.807 | 0.72 |
| | 7.11 | 1.007 | 1.26 | 4.95 | 0.817 | 0.83 |
| | 13.25 | 1.015 | 1.39 | 10.50 | 0.838 | 1.09 |
| | 18.50 | 1.02 | 1.47 | 15.45 | 0.855 | 1.25 |
| | 22.85 | 1.023 | 1.58 | 20.00 | 0.872 | 1.40 |

has been carried on with mixing impellers in batch systems of two immiscible liquids in an effort to find chemicals which will remain stable under repeated use such as must obtain in experimental work (*6, 7*). It was found that the ketone-water-acid system was stable over a period of at least two months, and reproducible drop size and coalescence rates could be obtained (*10*).

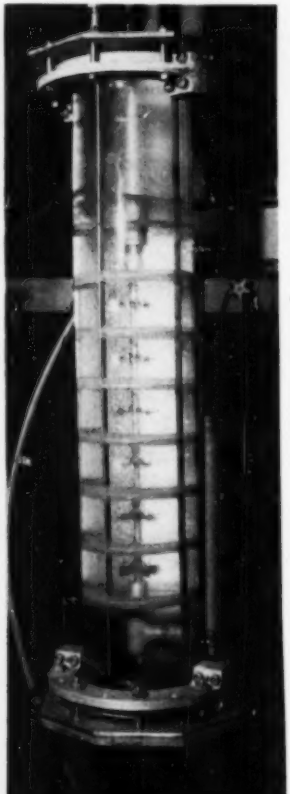

Fig. 1. Ketone flow up through water phase; no mixing. Interface above upper compartment.

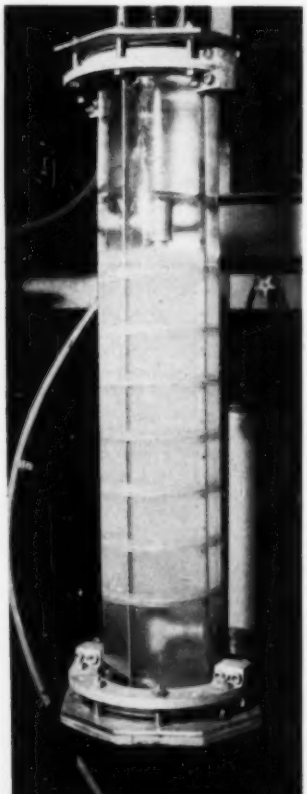

Fig. 2. Column in operation. Ketone flow up through water phase with mixing. Interface above upper compartment.

This ketone-water-acid system was chosen because others have used it in experiments with packed, spray, and agitated columns, and therefore direct comparisons could be made; and also because it is a system of sufficient stability that the ketone could be reused for a period of several weeks.

Batch experiments are also useful to compare drop size and coalescence characteristics for different-sized apparatus and work is now under way to correlate drop formation and coalescence in batch work with a change in equipment size. Batch tests have proven useful to predict the effect of such variables as flow rates, impeller speeds, and solute concentrations on column performance, and to scale-up to plant-size equipment with accuracy.

## Physical Properties, Equilibrium Data

**Methyl Isobutyl Ketone, Water, and Acetic Acid.** Equilibrium data for the system are available from Sherwood (*16*) for data at 77° F., Brinsmade and Bliss (*1*) at 77° and 111.8° F., Othmer, White and Treuger (*11*) at 73° F., and Scheibel (*15*) at 82° F. The present work was done at approximately 68° F., so the data just shown were examined to see whether the distribution ratio could be extrapolated to this temperature. A plot of the distribution ratio at constant water-phase acid concentrations against temperature shows that the data in the literature are not consistent with one another. Therefore, equilibrium data were redetermined.

Samples containing various amounts of the components, over an adequate range, were agitated for 30 min. and then placed in a thermostatically controlled bath overnight. Samples of each layer were then analyzed with either 0.1 or 1.0 normal sodium hydroxide (carbonate free) for acetic acid using phenolphthalein as an indicator. The ketone solutions were analyzed in alcoholic solution. Check samples analyzed with no alcohol also gave satisfactory agreement. All concentrations are given as weight per cent of total phase. The density of the phase was taken by means of a set of hydrometers. Viscosities were determined by using an Ostwald viscometer calibrated with distilled water.

These data, which are reported in Table 1, show that for dilute concentration ranges under 3 wt. % acid in the water layer, the equilibrium distribution ratio does not change appreciably over the range 68 + 5° F.; they agree fairly well with those of Scheibel and of Sherwood. Column experiments were therefore made at temperatures held between 64° and 73° F.

An attempt was made to get mutual solubility data by the method of Othmer, White, and Trueger (*11*). Considerable difficulty was experienced in getting accuracy in the dilute acid ranges. Calculations made from data taken by other investigators showed that for the concentration ranges used in the runs the operating curve was a straight line, and

thus the mutual solubility data were not needed. A sample of the ketone and water phases taken from the column at the end of the work yielded equilibrium data in perfect agreement with those listed in Table 1.

The interfacial tension of ketone-water-acid solutions is given by Cervi (*2*). He found that the addition of acetic acid decreases the interfacial tension from a value of 8.8 dynes/cm. at no acid to 3.0 dynes/cm. at a water layer concentration of 0.073 wt. %.

## Extraction Column

A photograph of the column is shown in Figure 1. It is constructed of a glass tube, four vertical baffles, horizontal plates with center holes, and impellers mounted on a shaft. The picture shows ketone flowing up from the bottom feed inlet, and the impellers are not rotating. Water feed at the top is carried down to the level of the top plate. There are seven plates shown, and they form six compartments. (To seal the plates to the uneven glass wall, pieces of Saran tubing can be seen inserted between the wall and plate). The interface is above the top plate. A wire screen was used to calm the settling zone. One screen can be seen above the interface, and another screen at the bottom of the column.

Figure *2* shows the column in operation with the impellers rotating and the ketone dispersed. The compartments are filled with circulating water and drops, and there is little intermixing between compartments. The two phases separate at a well-defined boundary a little above the top plate. There is no settling or coalescence between the compartments.

For scale-up information it is imperative that dimensionally similar systems and impellers be used (unless many more data are available), and it was desired to use impellers of at least 2-in. diam. so that they could conveniently be made dimensionally similar to commercial equipment. This set the minimum column diameter at about 6 in. since an impeller of 2-in. diam. is about the minimum for accurate scale model construction, and a column diameter about three times the impeller diameter is a nominal commercial selection figure. A 6-in. column could be used also with a 3-in. diam. impeller to observe the effect of impeller diameter.

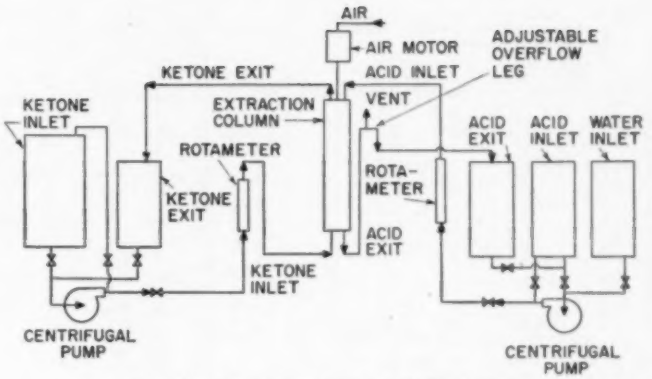


Fig. 3. Flow diagram for continuous liquid-liquid extraction.

Further details (*see* Fig. 3) of the column and auxiliary equipment are as follows:

A detail of the principal features of the column is given in Figure 4. It consisted of a 3-ft. length of 6-in. I.D. pyrex flanged glass pipe. The headers were of stainless steel. The stainless steel shaft was of 5/16-in. diam. The baffles were made from 1/8- by 1/2-in. stainless steel strip and the horizontal plates were of 1/16-in. stainless steel. Two sets of plates were used, one set having holes of 2 1/4-in. diam. and the other 3 1/4-in. diam. A Saran gasket was placed between the stator plate and the glass column to prevent by-passing of the liquid due to the irregular contour of the glass pipe. Twelve-mesh stainless steel screens were placed at each end of the column to calm the settling zones.

The feed stream was introduced through pipe tees concentric with the impellers. The take-off pipes are also shown in Figure 4.

Impellers were flat-blade turbines with proportions as shown in Figure 5. They were made of chromium-plated brass or of aluminum. The shaft was run by a Lightnin Mixer air motor.

The liquid lines were either 3/8-in. stainless steel pipe or Saran tubing. Saran was found to have a good resistance to methyl isobutyl ketone. The valves were of stainless steel.

The tanks were hard-rubber-lined (70 gal. capacity) except for one stainless steel tank (250 gal. capacity). The two pumps were Eastern Model, with Monel fittings.

## Operation of Column

To start a run, the column was allowed to fill with the continuous phase, and its flow was adjusted to the desired rate as shown by the rotameter. The disperse phase was then started and adjusted to the desired rate. In order that parallel operating and equilibrium lines could be used for calculation of results the ratio of the flow rates was held constant in the majority of runs. The centrifugal pumps discharged at high pressure so that the effect of a changing liquid head on the inlet side of a pump did not affect the flow rate appreciably for a given valve setting. Fine adjustments on the flow rates were made by using the by-pass lines of the pumps.

By raising or lowering the overflow leg on the water-solution exit the interface was adjusted to the desired height. In all the

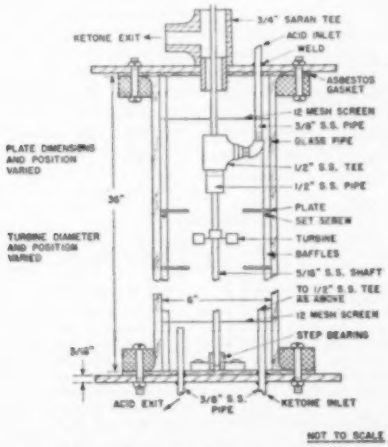


Fig. 4. Details of column construction.

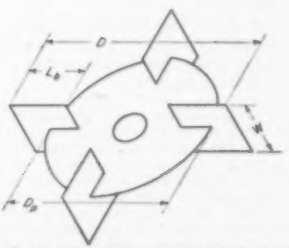


Fig. 5. Dimension ratios for standard flat-blade turbine.

$D : D_p : L_b : W = 20 : 15 : 5 : 4$

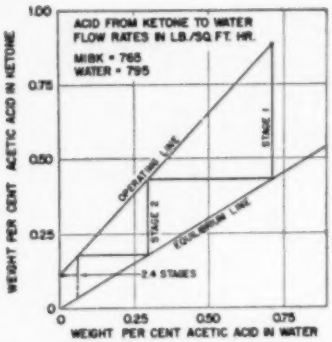


Fig. 6. Sample calculation for run No. 91.7; 4 turbines, 2-in. diam., 4 blades, position A; 4 stages, 4 in. high, 2½-in. diam. opening.

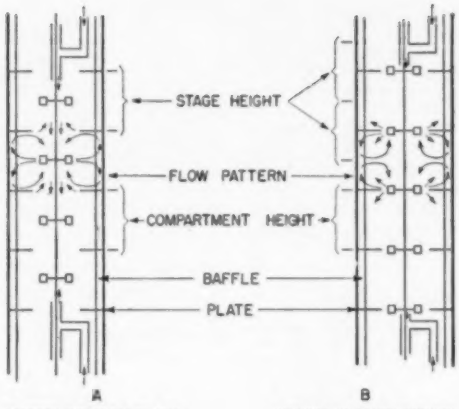
STAGE HEIGHT
FLOW PATTERN
COMPARTMENT HEIGHT
BAFFLE
PLATE
A
B
TURBINE CENTERED IN COMPARTMENT. STAGE COINCIDES WITH COMPARTMENT.
TURBINE AT PLATE OPENING. STAGE HEIGHT IS BOUND BY MID POINTS OF COMPARTMENTS. STAGE IS FORMED BY FLOW PATTERN.

Fig. 7. Impeller positions. Compartments formed by side wall and open plates.

runs water was the continuous phase, and the interface was held to within two or three inches above the top compartment, except of course, for flooding conditions.

The time to reach steady-state conditions was determined by taking 500-ml. samples of inlet and outlet streams at various intervals until the samples agreed with error of less than three per cent within themselves. This occurred at a total flow equal to about four times the column volume. After the first few runs, the column was allowed to operate until a total flow of from five to six times the column volume had passed through before samples were taken. For a given flow rate of both streams, several mixing speeds were used. The mixing speed was increased until a flooding condition was reached at which point the column would no longer operate satisfactorily.

Samples were analyzed by the same titration procedure as used to determine the equilibrium data. The temperature of the column was checked periodically; it was operated at room temperature, and always between 64° and 70° F.

The first seven variables listed earlier were investigated with dilute acid concentrations. To conserve acetic acid, the acid was extracted back and forth from the water and ketone phases. The runs when acetic acid was extracted from the water phase were used most often to correlate the variables, using a constant ratio of stream flow rates. Reverse extractions were used to investigate extreme conditions of flow rates and to determine the effect of the direction of solute travel.

The following hypothetical case of three runs, A, B, and C, illustrates the general procedure and concentrations used for the dilute runs.

In run A, the water for the inlet stream was made up to about 3% acetic acid by weight. The ketone inlet from the previous run contained about 0.5% acid by weight. The exit ketone, being high in acid, is used as the feed for the next run, B. In run B, pure water would be used as the solvent and would extract the acid from the ketone stream. The ketone would thus be dilute enough to be used in run C to extract acid from a 3% acid inlet. The water exit from run B would be discarded since it would contain only a small amount of acid. This procedure was followed, and the direction of solute extraction thus reversed from run to run.

In a brief study of the effect of solute concentration (variable 8), concentrations of acid up to 9% in the water phase were used.

## Evaluation of Results

Use of the height equivalent to a theoretical equilibrium contact stage gives a good physical concept of the performance of the column since the tower actually consists of compartmented stages. However, the tower could be thought of as operating in a differential countercurrent contact manner since the disperse phase travels progressively from compartment to compartment throughout the length of the column, and therefore, a height of a transfer unit (H.T.U.) or rate coefficient ($K_La$) calculation could be made. A qualitative observation made with a colored acid-base indicator showed that each compartment has a distinct and uniform concentration, and that the tower actually operated as a series of distinct stages. Color motion pictures were made of this operation. One disadvantage of correlating the over-all tower performance by H.T.U. or $K_La$ is that their values depend upon which phase is used for reference. Accordingly, the data here shown are given in terms of height equivalent to a theoretical stage (H.E.T.S.) and stage efficiency. Stage efficiency is found from the ratio of the number of equilibrium contacts computed from analytical results to the actual stages in the column. The number of equilibrium contacts was calculated graphically as illustrated in Figure 6.

The flow rates of the streams were adjusted to give approximately parallel operating and equilibrium lines so that the maximum accuracy could be achieved in the graphical calculations. If the equilibrium lines are straight and parallel, as was the case in much of this work, the over-all H.T.U. values for the individual phases and the height equivalent to a theoretical stage become identical (*5*).

Writing the material balances over a continuous countercurrent multistage

Table 2

Typical Data

| Run No. | Actual Stages | Stage Height, in. | Plate Opening, in. | Turbine Speed, rpm | Inlet Flow lb./hr.sq.ft. MIBK | Inlet Flow lb./hr.sq.ft. Water | Direction of Acid | Weight % Acetic Acid Inlet MIBK | Weight % Acetic Acid Inlet Water | Weight % Acetic Acid Outlet MIBK | Weight % Acetic Acid Outlet Water | Calculated Theoretical Stages | Stage Efficiency % | H.E.T.S., in. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 91.3 | 3 | 4 | 2-1/8 | 400 | 1350 | 795 | W → K | 0.282 | 2.12 | 0.956 | 0.973 | 2.13 | 71 | 5.7 |
| 103.2 | 3 | 4 | 3-1/4 | 300 | 1930 | 1100 | W → K | 0.417 | 1.93 | 0.900 | 1.07 | 1.99 | 66 | 6.0 |
| 113.4 | 4 | 3 | 2-1/8 | 375 | 1350 | 795 | W → K | 0.199 | 2.11 | 0.930 | 0.665 | 3.05 | 75 | 3.9 |
| 93.4 | 4 | 4 | 2-1/8 | 500 | 1350 | 795 | W → K | 0.242 | 1.89 | 0.845 | 0.635 | 3.18 | 79 | 5.0 |
| 97.7 | 4 | 4 | 2-1/8 | 400 | 1680 | 1010 | K → W | 0.895 | 0.00 | 0.168 | 1.08 | 3.44 | 86 | 4.7 |
| 121.2 | 4 | 6 | 2-1/8 | 350 | 1350 | 795 | W → K | 0.361 | 2.01 | 1.01 | 0.884 | 3.42 | 86 | 7.0 |
| 151.2 | 6 | 3 | 3-1/4 | 300 | 2360 | 1470 | W → K | 1.60 | 8.55 | 4.65 | 3.80 | 3.69 | 62 | 4.9 |
| 131.1 | 8 | 3 | 2-1/8 | 250 | 1350 | 795 | W → K | 1.79 | 9.76 | 5.43 | 3.14 | 6.60 | 82 | 3.6 |

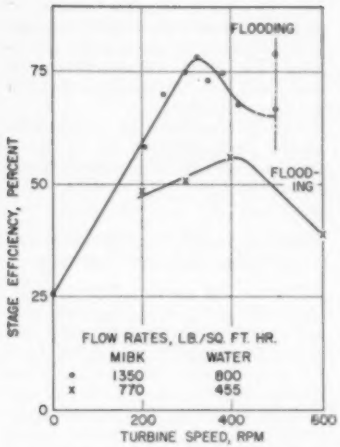


**Fig. 8. Effect of speed and flow rate on performance; 4 turbines, 2-in. diam., 4 blades, position A; 4 stages, 4 in. high, 2⅛-in. diam. opening.**

system, composition relationships between the streams are obtained. When partial miscibility exists, recourse can be had to graphical means to solve the equations.

Since the system is a three-component one, a triangular equilibrium diagram could be used. A convenient graphical method of stepping off stages was proposed by Varteressian and Fenske (*18*), and the operating line was found to be straight except for operating lines whose slopes differ by a factor of more than five with the slope of the equilibrium line. Sherwood used acid inlet concentrations of approximately 6 wt. %, about twice those used in this work. Since the slope of the operating and equilibrium lines in this work were within this range, it was possible to use a straight operating line and thus avoid the use of mutual solubility data.

The number of theoretical stages was calculated for each run. The height equivalent to a theoretical stage was calculated as the column height divided by the number of theoretical stages. The stage efficiency was obtained by dividing the theoretical stages by the actual number of stages. The column gave best performance when the impellers were centered in the compartments, and in this position a compartment coincided with an actual stage. (*See* Fig. 7, Position A.) The other impeller location is shown as Position B, and the height and location of a stage relative to a compartment is shown.

Extractions of acid were made both from ketone to water (abbreviated as K→W), and from water to ketone (W→K). The sample calculation of

Table 3

Summary of Data Tables

| Table No. | Number of Compartments | Number of Turbines | Number of Stages | Turbine Position (Fig. 7) | Height of Compartment, in. | Plate Opening Diam., in. | Turbine Diameter, in. | Number of Blades on Turbine | Low Acid Conc. | High Acid Conc. | Number of Runs |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 3 | 3 | 3 | A | 4 | 2-1/8 | 2 | 4 | X | | 16 |
| 5 | 3 | 3 | 3 | A | 4 | 3-1/4 | 2 | 4 | X | | 16 |
| 6 | 3 | 3 | 3 | A | 4 | 3-1/4 | 3 | 6 | X | | 24 |
| 7 | 4 | 4 | 4 | A | 3 | 2-1/8 | 2 | 4 | X | | 24 |
| 8 | 4 | 4 | 4 | A | 4 | 2-1/8 | 2 | 4 | X | | 29 |
| 9 | 4 | 4 | 4 | A | 6 | 2-1/8 | 2 | 4 | X | | 18 |
| 10 | 1 | 4 | 1 | A | 48 | complete | 2 | 4 | X | | 11 |
| 11 | 6 | 6 | 6 | A | 3 | 3-1/4 | 2 | 4 | X | X | 35 |
| 12 | 8 | 8 | 8 | A | 3 | 2-1/8 | 2 | 4 | X | X | 18 |
| 13 | 3 | 4 | 4 | B | 4 | 2-1/8 | 2 | 4 | X | | 29 |
| 14 | 3 | 4 | 4 | B | 4 | 3-1/4 | 3 | 6 | X | | 8 |

Figure 6 is for extraction from ketone to water. The same equilibrium curve was, of course, used for calculations in both directions, the operating line falling above the equilibrium line in the one case, and below in the other. Any error in the location of the equilibrium line data would be evident if wide discrepancies appeared in the extraction efficiencies for the different extraction directions. Data for both conditions give the same number of stages and efficiencies for comparable runs, and this gives added confidence in the accuracy of the equilibrium data.

All runs were checked by material balances, and only those which checked within ±10% were used.

## Tabulation of Results

Typical data for a few runs using the set-up shown in Figure 7A, with impellers located at the center of the compartments, are given in Table 2. The direction of diffusion is indicated, flow rates of both phases are shown as pounds per hour for a square foot of cross-sectional area of the column (for the 6-in. column the area is 0.196 sq.ft.), and rotational speed of the impellers is shown as revolutions per minute. Flooding is indicated by the letter *F* alongside the speed.

Compositions of the immiscible phases are given as weight per cent of acetic acid; these are used to set the operating

Table 4

Experimental Data

3 Stages, 4 in. High, 2-1/8 in. Plate Opening
3 Turbines, 2 in. Diameter, 4 Blades, Position A
F indicates flooding

| Run No. | Solute Direction | Inlet Flow Rate lb/hr.sq.ft. MIBK | Inlet Flow Rate lb/hr.sq.ft. Water | Turbine Speed rpm | Analyses Weight Per Cent Acetic Acid Inlet MIBK | Inlet Water | Exit MIBK | Exit Water | Theoretical Stages | Stage Efficiency % | H.E.T.S. IN. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 87.3 | W → K | 1350 | 1160 | 225 | 0.242 | 1.88 | 0.945 | 1.24 | 1.51 | 38 | 7.9 |
| 87.4 | | 1350 | 1160 | 380F | 0.242 | 1.88 | 1.04 | 2.16 | 2.31 | 72 | 5.2 |
| 91.1 | | 1350 | 795 | 200 | 0.282 | 2.12 | 0.905 | 1.06 | 1.70 | 57 | 7.1 |
| 91.2 | | 1350 | 795 | 310 | 0.282 | 2.12 | 0.970 | 0.898 | 2.45 | 83 | 4.9 |
| 91.3 | | 1350 | 795 | 400 | 0.282 | 2.12 | 0.956 | 0.973 | 2.12 | 71 | 5.7 |
| 91.4 | | 1350 | 795 | 650F | 0.282 | 2.12 | 0.928 | 0.990 | 1.94 | 65 | 6.2 |
| 89.1 | | 765 | 440 | 250 | 0.352 | 1.89 | 0.841 | 1.01 | 1.85 | 62 | 6.5 |
| 89.2 | | 765 | 440 | 350 | 0.352 | 1.89 | 0.865 | 1.03 | 1.86 | 62 | 6.5 |
| 89.3 | | 765 | 440 | 590 | 0.306 | 1.92 | 0.819 | 1.08 | 1.48 | 49 | 8.1 |
| 89.4 | | 765 | 440 | 750 | 0.306 | 1.92 | 0.815 | 1.08 | 1.45 | 48 | 8.3 |
| 89.5 | K → W | 1350 | 795 | 250 | 0.817 | 0.0 | 0.300 | 0.706 | 1.38 | 46 | 8.7 |
| 89.6 | | 1350 | 795 | 375 | 0.805 | 0.0 | 0.284 | 0.854 | 1.83 | 61 | 6.6 |
| 89.7 | | 1350 | 795 | 500F | 0.805 | 0.0 | 0.918 | 0.918 | 2.13 | 71 | 5.7 |
| 87.5 | | 765 | 795 | 250 | 0.910 | 0.0 | 0.531 | 0.531 | 1.06 | 35 | 9.4 |
| 87.6 | | 765 | 795 | 400 | 0.910 | 0.0 | 0.664 | 0.664 | 1.75 | 58 | 6.9 |
| 87.7 | | 765 | 795 | 600F | 0.910 | 0.0 | 0.672 | 0.672 | 1.82 | 61 | 6.6 |

Table 5

Experimental Data

3 Stages, 4 in. High, 3-1/4 in. Plate Opening
3 Turbines, 2 in. Diameter, 4 Blades, Position A
F Indicates Flooding

| Run No. | Solute Direction | Inlet Flow Rate lb/hr. sq. ft. | | Turbine Speed | Analyses Weight Per Cent Acetic Acid | | | | Theoretical Stages | Stage Efficiency | H.E.T.S. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Inlet | | Exit | | | | |
| | | MIBK | Water | rpm | MIBK | Water | MIBK | Water | | % | in. |
| 103.1 | W → K | 1930 | 1100 | 200 | 0.417 | 1.93 | 0.872 | 1.11 | 1.72 | 55 | 7.0 |
| 103.2 | | 1930 | 1100 | 300 | 0.417 | 1.93 | 0.900 | 1.07 | 1.99 | 64 | 6.0 |
| 103.3 | | 1930 | 1100 | 400 | 0.417 | 1.93 | 0.870 | 1.12 | 1.69 | 54 | 7.1 |
| 103.4 | | 1930 | 1100 | 750F | 0.417 | 1.93 | 0.863 | 1.12 | 1.61 | 52 | 7.4 |
| 101.1 | | 1350 | 795 | 200 | 0.242 | 2.09 | 0.813 | 1.03 | 1.46 | 47 | 8.2 |
| 101.2 | | 1350 | 795 | 300 | 0.242 | 2.09 | 0.845 | 1.04 | 1.51 | 49 | 7.9 |
| 101.3 | | 1350 | 795 | 400 | 0.242 | 2.09 | 0.875 | 1.05 | 1.59 | 51 | 7.6 |
| 101.4 | | 1350 | 795 | 650F | 0.242 | 2.09 | 0.840 | 1.06 | 1.48 | 48 | 8.1 |
| 101.5 | K → W | 1930 | 1100 | 250 | 0.860 | 0.0 | 0.429 | 0.733 | 1.02 | 32 | 11.7 |
| 101.6 | | 1930 | 1100 | 350 | 0.860 | 0.0 | 0.361 | 0.869 | 1.46 | 47 | 8.2 |
| 101.7 | | 1930 | 1100 | 450 | 0.860 | 0.0 | 0.330 | 0.900 | 1.58 | 51 | 7.6 |
| 101.8 | | 1930 | 1100 | 600F | 0.860 | 0.0 | 0.331 | 0.905 | 1.61 | 52 | 7.5 |
| 99.4 | | 1350 | 1160 | 250 | 0.803 | 0.0 | 0.308 | 0.508 | 1.00 | 33 | 12.0 |
| 99.5 | | 1350 | 1160 | 350 | 0.803 | 0.0 | 0.228 | 0.585 | 1.45 | 47 | 8.4 |
| 99.6 | | 1350 | 1160 | 450 | 0.782 | 0.0 | 0.208 | 0.605 | 1.60 | 52 | 7.5 |
| 99.7 | | 1350 | 1160 | 700F | | | | | | | |

Table 6

Experimental Data

3 Stages, 4 in. High, 3-1/4 in. Plate Opening
3 Turbines, 3 in. Diameter, 6 Blades, Position A
F Indicates Flooding

| Run No. | Solute Direction | Inlet Flow Rate lb/hr. sq. ft. | | Turbine Speed | Analyses Weight Per Cent Acetic Acid | | | | Theoretical Stages | Stage Efficiency | H.E.T.S. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Inlet | | Exit | | | | |
| | | MIBK | Water | rpm | MIBK | Water | MIBK | Water | | % | in. |
| 109.1 | W → K | 1930 | 1100 | 125 | 0.230 | 2.42 | 0.885 | 0.977 | 1.66 | 54 | 7.2 |
| 109.2 | | 1930 | 1100 | 200 | 0.230 | 2.42 | 0.870 | 0.950 | 1.61 | 52 | 7.4 |
| 109.3 | | 1930 | 1100 | 300F | 0.230 | 2.42 | 0.927 | 0.938 | 1.84 | 59 | 6.5 |
| 109.4 | | 1930 | 1100 | 250 | 0.230 | 2.42 | 0.926 | 0.920 | 1.87 | 61 | 6.4 |
| 105.1 | | 1350 | 795 | 150 | 0.265 | 2.14 | 0.917 | 1.06 | 1.71 | 57 | 7.0 |
| 105.2 | | 1350 | 795 | 200 | 0.265 | 2.16 | 0.937 | 1.08 | 1.71 | 57 | 7.0 |
| 105.3 | | 1350 | 795 | 250 | 0.265 | 2.16 | 0.917 | 1.08 | 1.65 | 55 | 7.3 |
| 105.4 | | 1350 | 795 | 300F | 0.265 | 2.16 | 0.907 | 1.13 | 1.52 | 51 | 7.9 |
| 107.1 | | 765 | 440 | 125 | 0.417 | 2.18 | 0.900 | 1.25 | 1.37 | 44 | 8.8 |
| 107.2 | | 765 | 440 | 200 | 0.417 | 2.18 | 0.920 | 1.25 | 1.42 | 46 | 8.4 |
| 107.3 | | 765 | 440 | 550F | 0.417 | 2.18 | 0.870 | 1.33 | 1.13 | 36 | 10.6 |
| 107.4 | | 765 | 440 | 300 | 0.417 | 2.18 | 0.892 | 1.31 | 1.23 | 40 | 9.8 |
| 105.5 | K → W | 1930 | 1100 | 150 | 0.886 | 0.0 | 0.465 | 0.835 | 1.10 | 36 | 10.9 |
| 105.6 | | 1930 | 1100 | 225 | 0.886 | 0.0 | 0.367 | 0.993 | 1.73 | 56 | 6.9 |
| 105.7 | | 1930 | 1100 | 300 | 0.886 | 0.0 | 0.336 | 1.01 | 1.89 | 61 | 6.4 |
| 105.8 | | 1930 | 1100 | 250 | 0.886 | 0.0 | 0.354 | 0.978 | 1.65 | 53 | 7.3 |
| 103.5 | | 1350 | 1160 | 155 | 0.861 | 0.0 | 0.313 | 0.610 | 1.20 | 39 | 10.0 |
| 103.6 | | 1350 | 1160 | 200 | 0.861 | 0.0 | 0.251 | 0.696 | 1.53 | 49 | 7.8 |
| 103.7 | | 1350 | 1160 | 250 | 0.885 | 0.0 | 0.239 | 0.706 | 1.62 | 52 | 7.4 |
| 103.8 | | 1350 | 1160 | 500F | 0.885 | 0.0 | 0.269 | 0.670 | 1.41 | 45 | 8.5 |
| 107.5 | | 765 | 795 | 130 | 0.765 | 0.0 | 0.281 | 0.281 | 0.95 | 32 | 12.6 |
| 107.6 | | 765 | 795 | 200 | 0.765 | 0.0 | 0.195 | 0.195 | 1.45 | 48 | 8.3 |
| 107.7 | | 765 | 795 | 460 | 0.765 | 0.0 | 0.202 | 0.202 | 1.32 | 44 | 9.1 |
| 107.8 | | 765 | 795 | 280 | 0.765 | 0.0 | 0.203 | 0.203 | 1.37 | 46 | 8.8 |

line (as in Fig. 6). The number of theoretical stages determined by graphical calculations are shown in the column titled Theoretical Stages. Stage efficiency and H.E.T.S. values are given in the last two columns.

Table 3 summarizes the conditions for all runs made and gives the number of the table wherein the complete results are tabulated.

## Effect of Solute Concentration and Direction of Solute Diffusion

The trend of correlations for impeller speed, diameter, and position, flow rate, and compartment dimensions was the same for both directions of solute travel and all acid-concentration levels. When a solute increases the solubility of two partially soluble liquids, the film resistance decreases and diffusion rate can be expected to increase with increasing solute concentration. This was found to be the case, the extraction efficiency being higher under identical operating conditions for higher acid-concentration levels. This is illustrated by the data of Table 15 which shows stage efficiency increasing with total acid content. This same phenomenon was noted by Johnson and Bliss (*8*) when using this system in a spray tower.

From the typical concentrations given in the procedure, it can be seen that the over-all acid concentration in the column is somewhat less when the acid is extracted from the ketone phase, since the inlet water contains no acid, than when the reverse extraction is performed. Extraction efficiencies were lower in that case, and the reason is ascribed to a lower over-all acid concentration in the column rather than the difference in the direction of solute diffusion.

Scheibel (*15*) also reports data for extracting acetic acid from both the organic phase and water phase. He reported a large difference in the efficiency for the two methods of extraction. In his work the water inlet concentrations when ketone was the extractant were about 20% and for the reverse operation were zero, making the difference in over-all acid concentrations quite large.

In the following correlations and curves for a given set of variables, approximately the same over-all acid concentration was used.

## Impeller Speed, Flow Rate, Flooding, Stage Efficiency

These variables are interconnected and must be discussed together. Typical plots of data are given in Figure 8, and show the operating characteristics of the column. At a constant flow rate the over-all stage efficiency goes up for an

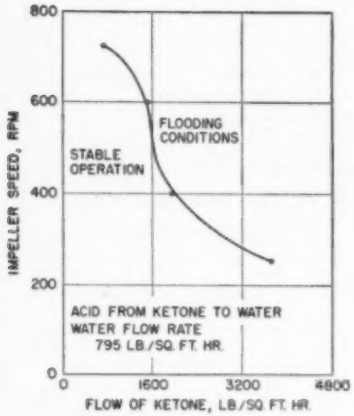


**Fig. 9. Effect of speed and flow rate on flooding; 4 turbines, 2-in. diam., 4 blades, position B; 4 stages, 4 in. high, 2¼-in. diam. opening.**

increase in speed, reaches a maximum, and then goes down as speed is increased further. Apparently too much agitation can cause some interstage mixing. Once this general behavior of the column had been established, succeeding runs were made by selecting speeds at constant flow rates to determine the maximum and the flood points for each flow rate.

For a particular flow rate, there is a definite impeller speed which produces flooding. Flooding in this column is similar to that observed in other types of columns. When the impeller speed was increased at a constant flow rate, a point was reached at which the holdup of the discontinuous phase suddenly became much greater. This caused the upper interface to rise, while a layer of discontinuous phase built up underneath the lower plate. The upper interface continued to rise, and the ketone backed up underneath the lower plate. Whenever the operation was continued the exit streams became contaminated with the opposite phase.

To relate column flooding to flow rate and impeller speed, the water rate was held constant and ketone rate varied until flooding was obtained at several speeds. (This was the only condition of operation which resulted in a deviation from parallel operating and equilibrium lines.) When the flow rate was increased in this way, flooding occurred at lower impeller speeds. This is illustrated in Figure 9, in which the flooding speed is plotted against flow rate of ketone. The region below the curve is a region of stable operation where flooding will not occur.

Since flooding depends upon two variables, throughput and impeller speed, it is difficult to say just what the maximum throughput would be. If a throughput is used which causes flooding with no agitation, the purpose of the column is defeated. Therefore, it was necessary to set up an arbitrary criterion for the maximum throughput at a particular compartment plate opening; one which allowed good agitation before flooding occurred. This maximum throughput, the optimum flow rate, is defined as the highest total flow rate for which the curve of stage efficiency vs. impeller speed shows a maximum point (Fig. 8). At flow rates higher than the optimum, the column will flood before the maximum point is reached. Typical flooding data are shown in Figure 10. Each curve is for a given flow. As the flow rate is increased from a low value in Curve 1, the efficiency of the column increases, and the speed to produce flooding goes down. Curve 3 is taken as the optimum

**Table 15**

**Effect of Overall Acid Concentration on Stage Efficiency**

Typical data are listed below:

6 compartments, 3 inches high, 3-1/4 inches opening between compartments.

6 turbines, 2 inches diameter, between plates, Position A.

Impeller speed, 400 rpm.

Flow rates, MIBK, 2080 lb./min.-sq.ft.
Water, 1160 lb./min.-sq.ft.

| Solute Direction | Acid Concentrations - Weight Per cent | | | | Stage Efficiency |
|---|---|---|---|---|---|
| | Inlet | | Outlet | | |
| | Water | MIBK | Water | MIBK | % |
| W → K | 9.0 | 1.47 | 3.34 | 4.55 | 58 |
| K → W | 0.0 | 4.88 | 5.06 | 1.32 | 52 |
| W → K | 2.84 | 0.57 | 1.36 | 1.34 | 42 |

**Table 16**

**Summary of Maximum Stage Efficiencies and Minimum H.E.T.S. Values in the Extraction of Acetic Acid from Water by Ketone. RPM between 300 and 450.**

| Turbine Diameter Inches | Turbine Position | Flow lb./hr.-sq.ft. MIBK | Flow lb./hr.-sq.ft. Water | No. of Stages | Stage Height Inches | Plate Opening Inches | Max. No. Theor. Plates | Min. HETS In. | Max. Stage % | Approx. Water Inlet conc. % |
|---|---|---|---|---|---|---|---|---|---|---|
| 2 | B | 1340 | 1160 | 4 | 4 | 2-1/8 | 2.0 | 8.0 | 50 | 3 |
| 2 | A | 1340 | 795 | 3 | 4 | 2-1/8 | 2.5 | 4.8 | 83 | 3 |
| 2 | A | 1340 | 795 | 4 | 4 | 2-1/8 | 3.15 | 5.1 | 79 | 3 |
| 2 | A | 1340 | 795 | 4 | 3 | 2-1/8 | 3.05 | 3.9 | 76 | 3 |
| 2 | A | 1340 | 795 | 4 | 6 | 2-1/8 | 3.40 | 7.0 | 86 | 3 |
| 2 | A | 760 | 490 | 4 | 3 | 2-1/8 | 2.4 | 5.0 | 60 | 3 |
| 2 | A | 1340 | 795 | 4 | 3 | 2-1/8 | 3.05 | 3.9 | 76 | 3 |
| 2 | A | 1340 | 795 | 8 | 3 | 2-1/8 | 4.7 | 5.1 | 59 | 3 |
| 2 | A | 1870 | 1040 | 4 | 4 | 6.0 | 1.45 | 11.1 | 36 | 3 |
| 2 | A | 1340 | 795 | 3 | 4 | 3-1/4 | 1.60 | 7.5 | 53 | 3 |
| 2 | A | 1870 | 1040 | 3 | 4 | 3-1/4 | 2.00 | 6.0 | 66 | 3 |
| 2 | A | 2360 | 1470 | 6 | 3 | 3-1/4 | 3.28 | 5.5 | 55 | 3 |
| 3 | A | 1340 | 795 | 3 | 4 | 3-1/4 | 1.70 | 7.0 | 57 | 3 |
| 3 | A | 1870 | 1040 | 3 | 4 | 3-1/4 | 1.90 | 6.3 | 63 | 3 |
| 3 | B | 1340 | 795 | 4 | 4 | 3-1/4 | 1.50 | 11.5 | 38 | 3 |
| 2 | A | 1350 | 795 | 8 | 3 | 2-1/8 | 6.00 | 4.0 | 75 | 9 |
| 2 | A | 2360 | 1470 | 6 | 3 | 3-1/4 | 3.69 | | 62 | 9 |
| Blank Run - No Plates or Agitation | | | | | | | | | | |
| 2 | A | 1870 | 1040 | 4 | 6 | 6.0 | 0.99 | 27.1 | — | 3 |

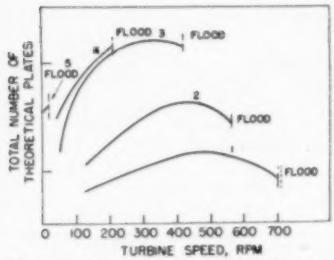


Fig. 10. Schematic diagram of determination of optimum flow rate. Flow rate increases from run 1 to run 5. Ratio of individual stream rates constant.

All other conditions constant.

Runs 1, 2—Low flow rates. Maximum efficiency present.

Run 3—Optimum flow rate.

Run 4—Floods before maximum point is reached.

Run 5—Floods at no agitation.

flow rate. At higher flow rates, Curve 4, the column will flood before the point of maximum efficiency is reached.

In many of the succeeding correlations the maximum stage efficiency (or minimum H.E.T.S.) obtained at a given flow rate is used. This maximum would be the high point on the curve of stage efficiency vs. speed, regardless of the impeller speed at which it occurred. Experimentally, it was not possible to arrive at the exact maximum point with an actual run, and therefore, the maximum point used here is the highest experimental point obtained, regardless of whether the curve indicates that the maximum might have been somewhat higher.

The maximum stage efficiency occurred within the speed range of 300 to 450 rev./min. for the 2-in. impeller. The exact speed in which the maximum occurred could not be determined without numerous experiments. Since over this range of 300-450 rev./min. the curve of efficiency vs. speed was fairly flat, the exact speed in which this mixing occurred is not considered in detail in succeeding correlations.

Table 16 summarizes the minimum H.E.T.S. and the maximum stage efficiencies obtained in the extraction of acetic acid from water solutions with ketone. Stage efficiencies listed are considered to be the best obtainable under the particular conditions of flow rate and column arrangement. By comparing runs which are identical except for one variable, the effect of that variable on the extraction efficiency may be found.

To determine maximum stage efficiencies for different operating conditions, four speeds were used at constant flow rate. These were a speed, close to, but lower than the maximum, a speed about 100 rev./min. higher than the maximum, a speed just at the flooding point, and the fourth speed at the predicted point of maximum stage efficiency.

In all cases, when the flow rate was increased, the stage efficiency also increased. This is illustrated in Figure 11, which shows the stage efficiency as a function of the total flow rate. The ratio of flow rates for the individual streams was held constant. No decrease of stage efficiency was noted for an increase in flow rate except when the column operated without any plates (Tables 10 and 16); then there was a leveling off at extremely high flow rates.

Data of Tables 10, 13, and 14, and in the summary of Table 16, show that the best position for the turbines is at the center of the compartments, shown as Position A in Figure 7. Plots of comparable data show Position A to give efficiencies in the order of one and one-half times those for Position B.

**Stage Height.** Maximum stage efficiency and minimum H.E.T.S. values are plotted as a function of stage height in Figure 12. It is seen that while the efficiency per stage goes up with increasing stage height, the H.E.T.S. values also go up. This is due to the fact that while the smaller stages are less efficient, more of them can be placed in a given tower height. Thus, the choice of the actual stage height to be used will depend upon whether the maximum efficiency per turbine is wanted, or the most theoretical stages in a given tower height. In any event, the minimum H.E.T.S. for a given throughput is the ultimate desire.

**Plate Opening.** Two sizes of compartment plate openings were used—2⅛ and 3¼ in. in diam. In addition, one set of runs was made without plates (*see* Table 3). At a particular flow rate, the stage efficiency of the 3¼-in. opening was considerably less than for the 2⅛-in. opening. This relation is shown in Table 17. However, the optimum flow rate is much larger for a 3¼-in. plate. Since the stage efficiency increases with increasing flow rate, the stage efficiency of the larger plate opening is increased to a value much closer to

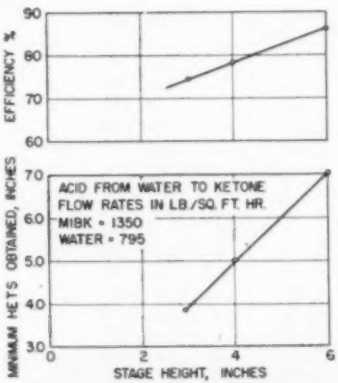


Fig. 12. Effect of stage height; 4 turbines, 2-in. diam., 4 blades, position A; stage height varied, 2⅛-in. diam. opening.

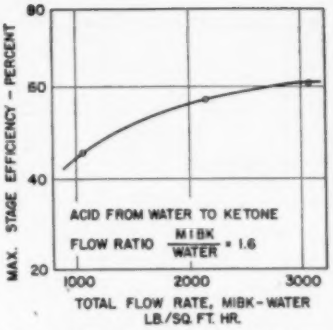


Fig. 11. Effect of flow rate on stage efficiency; 3 turbines, 3-in. diam., 6 blades, position A; 3 stages, 4 in. high, 3¼-in. diam. opening.

the stage efficiency for the smaller plate openings. The trend is clearly shown in Table 17. For example, at a flow rate of 2140 lb./(sq.ft.)(hr.), the 2⅛-in. plate openings gave a minimum H.E.T.S. of 4.9 in. while the 3¼-in. plate openings gave a minimum H.E.T.S. of 7.7 in. However, while 2140 is the optimum flow rate for the 2⅛-in. plate openings, the flow rate for the 3¼-in. plate openings can be increased to a value of 3940 lb./(hr.)(sq.ft.). This gives an H.E.T.S. of 6.0 in. for the larger opening, a considerable improvement over the lower flow rate.

The choice of large or small plate openings depends upon the relative importance of high column efficiency and/or high column throughput.

**Impeller Diameter.** A 3-in. flat-blade turbine was used to compare with the 2-in. turbine used in other correlations. The value of the stage efficiency for the 3-in. turbine at a given flow rate was within ±5% of the value for the 2-in. turbine.

Since large turbines will draw more power than small turbines at equal speeds, it is of interest to compare the two turbines in regard to power input at the maximum stage efficiency. The maximum stage efficiency for the 3-in. turbine occurred at a lower speed than for the 2-in. turbine as shown in Tables 6 and 7. Since the maximum stage efficiency does not occur at a well-defined speed, direct comparison between the power consumption of the two turbines is not conclusive.

Comparing the data from Tables 6 and 7, there is a wide variation between the ratio of power drawn by the 3-in. turbine and power drawn by the 2-in. turbine at the maximum stage efficiency, although taking an average of the ratios gives a value of approximate unity. Countercurrent extraction does not give conclusive comparison between performance of different ratios of impeller to tank diameter. In most mixing operations, there is an optimum ratio of impeller to tank diameter to achieve a given result.

**Total Number of Stages.** Since the 3-in. plate spacing gave lower H.E.T.S. values than the larger spacings, it was decided to place eight mixing compartments in the 3-ft. column. Results appear in Table 12. The over-all efficiency was somewhat less than for three and four stages. It appears, however, that the over-all acid concentration has a definite effect which may mask the effect of number of stages. For example, Table 16 shows the

Table 17

Effect of Size of Opening
Between Compartments

Typical data for operation with:

4 stages, 4 inches stage height
4 turbines, between the plates, Position A
Extraction, W → K.
Low Acid Concentrations

| Compartment Opening | Maximum Stage Efficiency | Minimum H.E.T.S. Inches | Flow Rate |
|---|---|---|---|
| | | | Constant |
| 0 | 100 | 4 | 0 ⚡ |
| 2-1/8 | 83 | 4.9 | 2140 ⚡ |
| 3-1/4 | 52 | 7.7 | 2140 |
| 6 | 36[1] | 10.5 | 2140 |
| | | | At Maximum Efficiency |
| 0 | 100 | 4.0 | 0 ⚡ |
| 2-1/8 | 83 | 4.9 | 2140 ⚡ |
| 3-1/4 | 67[2] | 6.0 | 3950 ⚡ |
| 6 | 36[1] | 10.5 | 4400 ⚡ |

⚡ Optimum flow rate.
(1) Data taken for reverse extraction corrected to W → K.
(2) Data taken for 6 stages, 3 inches high, corrected to 4 stages, 4 inches high.

maximum efficiency for the eight-stage column to be 75% for 9% acid concentration in the water inlet, and only 59% efficiency for 3% acid in the water inlet. Hence, it is felt that the efficiency is affected by drop size occasioned by interfacial tension due to acid concentrations, as well as by the effect of turbine speed on drop size. The Weber number may well prove to be a necessary parameter to evaluate this phenomenon.

The larger the degree of separation performed in a column, the greater will be the difference in the physical properties of each phase at the top and bottom stages. This should cause a difference in the bubble size produced in the various stages. The column operation as far as settling is concerned, is limited by the smallest size particle in any stage. Thus, one stage may have the optimum degree of dispersion while the others may have a lower efficiency. There is also a tendency for more and more refinement and entrainment of the droplets as they pass through the column. If this effect is increased by the changes in fluid properties, a considerable difference in drop size should be noticed.

Visually, a definite decrease in the bubble or drop size was noticed from the bottom to the top of the column. Since the top stage had the finer bubbles, this limited the column operation from the point of continuous settling and flooding. This effect was also noticed in the four-stage setup, but only to a slight degree. The effect was most pronounced for the runs using a pure water inlet.

**Process Power Requirements.** Power requirements can be estimated using the data of Rushton, Costich and Everett (*13*) since the impellers used were dimensional homologues. For Reynolds numbers greater than 10,000 the power for each turbine operating in arrangement A, Figure 7, can be calculated from

$$P = \frac{K}{g}\rho N^3 D^5$$

where $P$ is power in ft.lb./sec.,

constant $K$ is 4.4 for the four blade turbines

$g$ is gravity constant

$\rho$ is average density of mixed liquids

$N$ is turbine speed in rev./sec.

$D$ is turbine diameter in ft.

## Comparisons with Other Types of Columns

To compare the results from this column with those from other types of extraction towers, it is best to use results with the same chemical system and the same over-all acid concentration, and the same column diameter. Data are available for the same chemical system, but these are not exactly comparable data for the same diameter column.

The highest efficiency reported by various investigators using the system methyl isobutyl ketone, acetic acid and water are given in Table 18. When comparing the data, note that when the acid concentration is raised, the column efficiency increases (thus decreasing the H.E.T.S.). Increasing the column diameter increases the H.E.T.S. in most mass-transfer operations.

The data show that the present column has an extremely high efficiency compared with conventional packed and spray towers at moderate throughput rates. For example, the H.E.T.S. of this column is of the order of one-half to one-third the values for packed towers (Table 18). The throughput of the packed tower is somewhat greater. However, packed towers (*3*) are not usually operated close to the flooding point, since the maximum efficiency occurs at a lower throughput. Since no packing is used in this column, it is easier to keep clean, and the design of the intermediate settling or coalescing zones is eliminated. The column is also extremely flexible with regard to variable throughput and impeller speeds. The maintenance of packing glands and bearings for the moving parts should not be a significant problem, except possibly for high pressure work, or under corrosive conditions.

In regard to scale-up to larger sizes, comparison of batch runs in a 6-in. and in a 12-in. tank with methyl isobutyl ketone water acetic acid has indicated that no unusual problems are present and it is thought that the application of standard scale-up techniques now in use for designing mixer applications will be

Table 18

Comparison of Typical Results of High Capacity Extraction Towers
Using the System Methyl Isobutyl Ketone, Water and Acetic Acid

The details of column performance listed below are the best results reported under the operating conditions investigated.

| Type of Column | Diameter of Column Inches | Details of Column | Approx. Conc. of Inlet water Stream | Minimum H.E.T.S. for W → K | Throughput total stream lb/hr./ft.² hr. |
|---|---|---|---|---|---|
| Multiple Mixing compartments this work | 6 | 4 stages, 3 in. high, 2-1/8 in. opening, 2 in. turbines, centered in compartments. | 3% | 3.9 | 2140 ⚡ |
| Multiple Mixing compartments this work | 6 | 8 stages, 3 in. high, 2-1/8 in.opening, 2 in. turbines, centered in compartment. | 3% | 5.1 | 2140 ⚡ |
| Multiple Mixing compartments this work | 6 | 8 stages, 3 in. high, 2-1/8 in. opening, 2 in. turbines, centered in compartments. | 9% | 3.7 | 2140 ⚡ |
| Multiple Mixing compartments this work | 6 | 6 stages, 3 in. high, 3-1/4 in. opening, 2 in. turbines, centered in compartment. | 9% | 5.5 | 3950 ⚡ |
| Scheibel (13) | 1 | 13 stages, 1/2 in. mixing sect., 2 in. packing ht. | 23.0% | 2.2 | 1340 |
| Scheibel (14) | 12 | 3 stages, 3 in. mixing sect., 9 in. packing ht. | 28.1% | 9.0 | 4200 |
| Packed (15) | 3.93 | 66 ins. high. | 6.0% | 10.0 | 6300 |

⚡ Optimum. Higher Throughputs can be obtained but stage efficiencies are lower.

Note: A comparison of packed, perforated plate, and spray towers under identical operating conditions for the system toluene-water-benzoic acid was made by Row, Koffolt, and Gilmore (12). The ratio of H.E.T.S. values was approximately. Packed:perforated plate:spray::2:3:10. The throughputs were in the inverse ratio, the spray tower having the highest throughput.

satisfactory. The scale-up of mixers in heat- and mass-transfer reactions has been solved on many commercial applications by either empirical methods or the application of dimensional analysis. Once data on a column size different from 6 in. are available, it will be possible to see how column performance is affected by column size, and the operating and economic problems involved in the construction of large columns can be evaluated.


## Acknowledgment

This work was supported by a fellowship from the Mixing Equipment Co., Rochester, N. Y., for basic research in the chemical engineering laboratories of Illinois Institute of Technology.


## Notation

$D$ = turbine diameter, ft.
$D_p$ = diameter of turbine disc
$F$ = flooding
$g$ = gravitational constant
H.E.T.S. = height equivalent to a theoretical stage
H.T.U. = height of a transfer unit
HAc = acetic acid
$K$ = constant in equation;

$$P = \frac{K}{g}\rho N^3 D^5$$

$K_L a$ = capacity coefficient, liquid
$K \rightarrow W$ = symbol for direction of solute travel, solute from ketone to water phase
$L$ = liquid flow rate, either phase
$L_b$ = turbine blade length
$N$ = turbine speed, rev./sec.
MIBK = methyl isobutyl ketone
$P$ = power (ft.)(lb. force)/(sec.)
$W \rightarrow K$ = direction of solute travel, from water to ketone phase
$\rho$ = density, lb./cu.ft.
$\mu$ = viscosity, lb./(sec.)(ft.)

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## Discussion

**Lawrence Lynn** (Celanese Corp., Clarkwood, Tex.): You showed how this mixer-column compared with the Scheibel extractor. How would it compare with a Podbielniak column or a packed column unit of about the same size? How would it work as far as the efficiency as well as throughput is concerned?

**J. Y. Oldshue:** It is rather hard to compare the mixer-settler unit reported here with the centrifugal type. The field of application for the centrifugal extractor is primarily where the phases are particularly hard to separate and a low solvent holdup is necessary. You get a large number of stages in the centrifugal type, but the capacity of a single unit is limited to the mechanical design of the rotor and mechanical seals. I feel that the mixer tower can have much larger capacities than the centrifugal type. Our information indicates that there would be no particular trouble in scale-up but I must emphasize that we have not tried a plant-size column.

**Lawrence Lynn:** Is 6 in. the largest size you've tried in your work to date?

**J. Y. Oldshue:** Yes. We have run batch experiments in both 6-in. and 12-in. tanks to get some idea of the nature of the settling rate, and based on these studies we see no particular trouble in scaling-up, but we have not tried any columns larger than 6 in.

**Lawrence Lynn:** In scaling-up power requirements, I presume that you can use the general mixing equations.

**J. Y. Oldshue:** That is correct. We have data on the power consumption of all our impellers as a function of impeller speed and fluid properties. The power required to accomplish a particular operation is not always available in a mathematical form, but long experience in the scale-up of mixing operations has given us considerable mathematical and empirical information.

**Lawrence Lynn:** Would you have any idea about the cost of the commercial size 8-ft. columns compared with the present Scheibel columns that have been advertised in the recent literature?

**J. Y. Oldshue:** I think the two columns would be comparable in cost since both units require vertical shafts passing through the column and an agitator drive. In our columns we have eliminated the packing so that cost would not be present.

**Lawrence Lynn:** You didn't show any tray compartment details in the slides. Do you have simply rings or are there rings with jets?

**J. Y. Oldshue:** The plates separating compartments are merely circular rings with circular holes in the center.

**R. W. Welborn** (B. F. Goodrich Research Center, Brecksville, Ohio): Would you care to comment on the sort of minimum residence times that can be obtained on the discontinuous phase in your column in the middle range of stage efficiencies?

**J. Y. Oldshue:** An average value would be about 5 min. for a complete column turnover with a column 24 in. high and of 6-in. diam.

**Lawrence Lynn:** Have you ever tried to use your extractor on a concentrated solution of acetic acid rather than dilute feed stock? We have been led to understand that with the Scheibel column you get an entirely different type of extraction from that shown.

**J. Y. Oldshue:** Practically all our runs were done at less than 3% acetic acid in the incoming stream. The brief study we did make of solute concentration was done with 9% acetic acid.

**A. W. Geers** (Du Pont Co., Wilmington, Del.): You mentioned the interface locations at the top and bottom of the column, yet you did not discuss operation with the interface located at the bottom. Did you operate with the interface at the top only?

**J. Y. Oldshue:** Our operation was entirely with water the continuous phase and the interface at the top. There is, of course, a separation between the dispersed phase and the clear continuous phase leaving the column from the lower stage.

**L. O. Frescoln** (Hercules Powder Co., Wilmington, Del.): What sort of height over diameter ratios would you use for different size columns in the individual mixing chambers?

**J. Y. Oldshue:** We would probably use the compartment height about ½ the column diameter.


*(Presented at A.I.Ch.E. Kansas City (Mo.) Meeting.)*

# A CORRELATION OF SOLIDS TURNOVER IN FLUIDIZED SYSTEMS

## *Its Relation to Heat Transfer*

MAX LEVA† and MILTON GRUMMER

Bureau of Mines, Bruceton, Pennsylvania

**Vertical velocities of particles along the container wall were determined in fluidized sand and silica gel beds. Particle velocities were then correlated in terms of quantities that are readily observed and calculated for fluidized beds. The correlation, based on the energy exchange between the fluidizing gas and the solids led to the definition of a new concept, the "inter-particle" friction factor.**

**Film coefficients of heat transfer between fluidized beds and the surroundings were analyzed and it was shown that for the type of equipment considered film coefficients increased with the 0.6 power of the particle velocity. A brief survey of other published data showed a similarity between various methods of correlation. Results indicated that a correlation of heat-transfer data in terms of particle velocities should lead to a universal correlation, but before such a generalization was possible a better understanding of the effect of special equipment features on the dynamics of the particles in the charge was required.**

RECENT investigations of heat transfer in fluidized beds have shown (*2, 5, 6, 10-12*) that the presence of fluidized solids increases heat-transfer rates materially over what would be expected in empty pipes or fixed packed beds, operating under the same rate of mass flow. The usual explanation has been that, because of the motion of the particles past the inside of the confining wall, the thickness of the fluid film is decreased, thus reducing its thermal resistance. This explanation is supported by the work of Baerg, Klassen, and Gishler (*2*), who investigated heat flow in fluidized beds, and also obtained limited data prior to fluidization. As mass velocities reached minimum fluidization values a sharp increase in film coefficients of heat transfer was noted.

**Experimental.** The dynamic pattern of particle motion in fluidized charges of particles having relatively small diameters previously described is shown in Figure 1. In all cases it was reported that particles descended along the wall, and ascended fountain-like further from the wall, especially in the center of the bed. Numerous vortices were also observed throughout the entire column. These probably originated from a horizontal velocity component of the particles, not considered in the present analysis. The vortices will influence particle mixing by a "dilution effect." The net effect is a gradual revolution of material through the column, i.e., a turnover of solids. The material does not move "in bulk," rather it is gradually diluted by intruding particles (as a consequence of the vortex effect) until a complete turnover is eventually achieved.

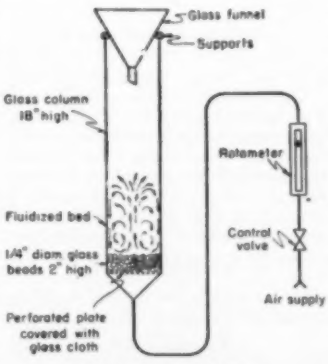


Fig. 1. Experimental unit.

A suitable tracer material was necessary for observation of the average particle velocity. As the physical properties of the tracer should be those of the charge, a minute quantity of gentian violet or fuchsine dye was precipitated on a portion of the solids to be tested. This procedure limited the choice of materials to those which could be dyed well enough to be clearly differentiated, when highly dispersed, from the undyed particles in the column. Silica gel absorbed the dye readily from alcoholic solution. Sand could be dyed only by making it into a paste with a strong alcoholic solution of dye and evaporating the solvent. Ground alundum and a Fischer-Tropsch iron catalyst could not be dyed or otherwise readily differentiated from the main portion of the solids. When white sand was used with beds of black catalyst or coal, such large quantities of tracer were required for satisfactory observation that the characteristics of the bed were believed to be strongly influenced by the addition of foreign matter.

Preliminary experiments indicated that solids with appreciable channeling tendencies gave nonreproducible results. This further limited the choice of materials to particles generally larger than 325-mesh. Activated alumina particles, which accepted dyes readily, channeled too severely to give consistent results. Data pertaining to the materials investigated are shown in Tables 1-3. Most charges were close cuts, but a few mixtures were also tested. Experiments were conducted in 1.32- and 2.032-in. diam. tubes. For any particular run, the vessel was charged with a definite quantity of the undyed material, and air was admitted at a sufficient rate to assure good solids circulation. Without changing the air rate, a definite quantity of the tracer was introduced through a funnel which was gently rotated to distribute the colored material evenly over the cross section of the tube. The time interval between addition of tracer and the establishment of visual uniformity of the colored material through the entire bed was then measured with a stop watch. The difficulties of distributing the tracer uniformly over the column cross section limited the investigation to tubes of the comparatively small sizes just indicated.

Complete original data are on file (Document 2676) with American Documentation Institute, 1719 N Street Northwest, Washington 6, D. C. Obtainable by remitting $0.50 for microfilm and $1.00 for photocopies.

† Present address: Consulting chemical engineer, Pittsburgh, Pa.

Table 1.- Packing characteristics.

| Experiment | Material | $D_p$ (inch) | Packing uniformity |
|---|---|---|---|
| a/ | Round sand | 0.00892 | Uniform |
| b | Mixed round and sharp sand | .00514 | Mixture |
| c | Round sand | .00340 | Mixture |
| d | Round sand | .00964 | Uniform |
| e | Round sand | .01505 | Uniform |
| f | Sharp sand | .01785 | Uniform |
| g | Sharp sand | .00995 | Mixture |
| h | Sharp sand | .00333 | Mixture |
| i | Silica gel | .00345 | Uniform |
| j | Silica gel | .00488 | Uniform |
| k | Silica gel | .01268 | Uniform |
| l | Silica gel | .01062 | Mixture |
| m | Silica gel | .00700 | Mixture |
| A | Round sand | .01062 | Uniform |
| B | Sharp sand | .00326 | Mixture |
| C | Silica gel | .01057 | Uniform |

a/ Experiments a to m were performed in small tube; A to C in large tube.

Table 2.- Size distribution of mixtures investigated.

| Experiment | Mesh size | | | | | | | | | | $D_p$ (inch) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | <100 | 100-135 | 135-150 | 150-200 | 200-250 | 150-200 | 170-200 | 80-170 | 60-170 | >60 | |
| a | - | - | 1.2 | 4.5 | 27.5 | 37.5 | 14.4 | 12.9 | - | - | 0.00514 |
| c | - | 0.5 | 1.3 | 6.2 | 85.5 | 6.1 | - | - | - | - | .00340 |
| g | - | - | - | - | - | - | 3.5 | 31.5 | 62.0 | 3.0 | .00995 |
| h | 1.2 | 5.0 | 12.2 | 15.0 | 45.2 | 20.4 | - | - | - | - | .00333 |
| l | - | - | - | - | - | 1.3 | 3.2 | 27.2 | 41.5 | 26.0 | .01062 |
| m | 1.5 | 4.5 | 7.0 | 8.0 | 4.5 | 16.5 | 6.7 | 18.2 | 31.5 | 3.0 | .00700 |

The uncertainty as to whether the tracer was actually dispersed uniformly in the bed at the end of the mixing period (since only dispersion on the wall could be observed) was eliminated by examining portions of the bed interior.

Orienting experiments showed that addition of small quantities of tracer produced so small a final concentration of tracer through the column that precise detection was difficult. The accuracy was best with about 10% of tracer material. With larger amounts distribution difficulties resulted, and streaks were frequently observed, which reached the bottom of the column sooner than other portions of the tracer. This was often the result of an unequal initial distribution on top of the column.

Attempts were made to introduce the tracer into the base of the column, below the colorless material. This method required a rapid adjustment of flow, and, as small differences in packing density between tracer and colorless charge could not be avoided, immediate, smooth fluidization was impossible. The method was abandoned as unworkable.

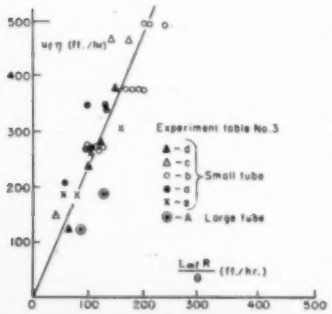


Fig. 2. Round sand data.

**Correlation of Data.** The assumption is made that in a fluidized bed of differential height, fluidization efficiency (7) and particle movement are those of the entire bed. If the horizontal velocity components of the particles in the bed may be considered negligibly small when compared with the vertical velocity components, the energy exchange occurring between the fluid and the moving particles in the bed may be expressed by:

$$u_f A_T \eta d\Delta p = \beta s d w \qquad (1)$$

which on integration between the limits $0 \rightarrow \Delta p$ and $0 \rightarrow w$ becomes:

$$u_f A_T \eta \Delta p = \beta s w \qquad (2)$$

Since for solid-gas systems $A_T \Delta p \simeq w$, Equation (2) becomes:

$$u_f \eta = \beta s \qquad (3)$$

The vertical particle velocity may be observed and is represented by:

$$s = \frac{L_{mf} R}{\theta}$$

substituting into Equation (3),

$$u_f \eta = \beta \frac{L_{mf} R}{\theta} \qquad (4)$$

The original and calculated data are reported in Table 3. If a correlation patterned similar to Equation (4) is possible, a plot of the calculated product $u_f\eta$ vs. the observed quantity $L_{mf}R/\theta$ should on cartesian coordinates yield a straight line, passing through the origin and having a slope numerically equal to the constant $\beta$. In Figures 2, 3 and 4 the data for round sand, sharp sand and silica gel have been presented in this manner.

Values of $\beta$ are 2.55 and 2.25 for the sharp and round sands and 1.15 for the silica gel. It is not surprising that the values differ for the different materials. To interpret the nature of $\beta$ and in particular its dependence on the material under consideration, data from experiments $A$, $g$ and $l$ are shown in Figure 5. The solids used in these runs are of approximately the same composite diameter and therefore have approximately the same fluidization efficiencies for a given mass velocity. The figure shows $u_f$ in relation to $L_{mf}R/\theta$, and apparently for any linear air velocity, e.g., 1200 ft./hr., the sharp sand has the smallest value of $L_{mf}R/\theta$, the round sand has the next larger, and the silica gel has the largest value by far. As expansion ratios of the beds differ relatively slightly from each other the time required for mixing the upper 10% of solids uniformly into the bed diminishes from sharp to round sand and then to the gel. Mixing pro-

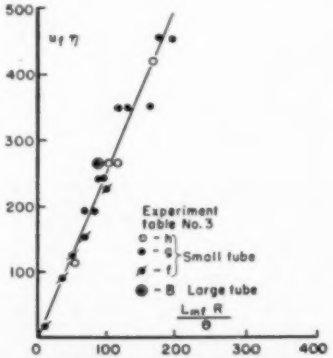


Fig. 3. Sharp sand data.

Table 3.— Original and calculated data.

| Run number | Weight of charge, grams | $u_f$ ft./hr. | seconds | | R | $L_{mf}R/\theta$ (ft./hr.) |
|---|---|---|---|---|---|---|
| a-1 | 100 | 1173 | 15 | 0.192 | 1.153 | 57.6 |
| a-1 | 100 | 1169 | 8 | .296 | 1.212 | 137 |
| a-1 | 100 | 1293 | 30 | .230 | 1.185 | 107 |
| a-1 | 100 | 1283 | 11 | .230 | 1.185 | 97.7 |
| a-2 | 200 | 1169 | 29 | .296 | 1.212 | 99.4 |
| a-3 | 150 | 1169 | 12 | .296 | 1.212 | 137 |
| b-1 | 100 | 652 | 10 | .449 | 1.168 | 118 |
| b-1 | 100 | 652 | 12 | .449 | 1.168 | 98 |
| b-1 | 100 | 652 | 9 | .449 | 1.168 | 131 |
| b-1 | 100 | 798 | 7 | .372 | 1.193 | 172 |
| b-1 | 100 | 798 | 6 | .372 | 1.193 | 201 |
| b-1 | 100 | 957 | 5 | .319 | 1.236 | 247 |
| b-2 | 150 | 957 | 9 | .319 | 1.236 | 206 |
| b-2 | 150 | 798 | 10 | .372 | 1.193 | 181 |
| b-2 | 150 | 652 | 15.6 | .449 | 1.168 | 113 |
| b-2 | 200 | 957 | 11.5 | .319 | 1.236 | 215 |
| b-3 | 170 | 798 | 10 | .372 | 1.193 | 205 |
| c-1 | 100 | 644 | 7.5 | .701 | 1.397 | 157 |
| c-1 | 100 | 543 | 7 | .699 | 1.374 | 186 |
| c-1 | 100 | 275 | 27.5 | .520 | 1.200 | 48.2 |
| c-2 | 150 | 644 | 9 | .724 | 1.397 | 196 |
| d-1 | 100 | 405 | 9.5 | .684 | 1.138 | 123 |
| d-1 | 100 | 231 | 17.5 | .598 | 1.065 | 63.5 |
| d-2 | 65 | 361 | 7.5 | .661 | 1.119 | 101 |
| e-1 | 100 | 3060 | 19 | .0607 | 1.231 | 53.5 |
| e-1 | 100 | 4040 | 11 | .0660 | 1.330 | 100 |
| e-1 | 100 | 4550 | 7 | .0674 | 1.371 | 142 |
| e-1 | 100 | 3550 | 14 | .0526 | 1.282 | 76 |
| f-1 | 100 | 3042 | 25 | .0301 | 1.208 | 45.4 |
| f-1 | 100 | 3508 | 19 | .0456 | 1.263 | 62.6 |
| f-1 | 100 | 3985 | 15 | .0574 | 1.306 | 81.6 |
| f-1 | 100 | 4475 | 10 | .0728 | 1.352 | 127 |
| f-2 | 200 | 3508 | 134 | .0178 | 1.263 | 17.6 |
| g-1 | 100 | 1168 | 25 | .130 | 1.186 | 82.0 |
| g-1 | 100 | 1038 | 24 | .148 | 1.221 | 90.0 |
| g-1 | 100 | 1990 | 16 | .175 | 1.292 | 133 |
| g-1 | 100 | 2265 | 8 | .200 | 1.358 | 176 |
| g-2 | 150 | 1990 | 17 | .175 | 1.292 | 119 |
| g-3 | 200 | 1668 | 28 | .130 | 1.186 | 68.3 |
| g-3 | 200 | 1638 | 13 | .136 | 1.221 | 97.0 |
| g-3 | 200 | 1990 | 8 | .175 | 1.292 | 167 |
| g-3 | 200 | 2265 | 7 | .200 | 1.358 | 200 |
| h-1 | 100 | 274 | 84 | 0.425 | 1.200 | 53.7 |
| h-1 | 100 | 451 | 13 | .589 | 1.369 | 105 |
| h-1 | 100 | 627 | 8 | .671 | 1.701 | 167 |
| h-1 | 100 | 451 | 11.5 | .589 | 1.369 | 119 |
| i-1 | 50 | 231 | 8 | .579 | 1.313 | 131 |
| i-1 | 50 | 176 | 15 | .512 | 1.218 | 60.4 |
| i-2 | 100 | 231 | 16 | .579 | 1.313 | 137 |
| j-1 | 50 | 231 | 13.5 | .354 | 1.110 | 72 |
| j-1 | 50 | 363 | 10 | .491 | 1.178 | 163 |
| j-1 | 50 | 405 | 6.5 | .519 | 1.197 | 161 |
| j-2 | 100 | 405 | 15 | .519 | 1.197 | 160 |
| j-3 | 75 | 405 | 9 | .519 | 1.197 | 158 |
| k-1 | 50 | 957 | 15 | .059 | 1.160 | 77.2 |
| k-1 | 50 | 1173 | 12 | .069 | 1.216 | 188 |
| k-1 | 50 | 1167 | 7 | .100 | 1.303 | 186 |
| k-2 | 100 | 1167 | 21 | .100 | 1.303 | 128 |
| k-3 | 75 | 1167 | 11.5 | .100 | 1.303 | 169 |
| l-1 | 50 | 795 | 10 | .099 | 1.249 | 107 |
| l-1 | 50 | 956 | 20 | .121 | 1.227 | 113 |
| l-1 | 50 | 1173 | 7 | .144 | 1.276 | 167 |
| l-1 | 50 | 1282 | 5 | .158 | 1.310 | 252 |
| l-2 | 100 | 1282 | 15 | .158 | 1.310 | 176 |
| m-1 | 50 | 361 | 10 | .711 | 1.108 | 97.9 |
| m-1 | 50 | 361 | 9 | .211 | 1.108 | 103 |
| m-2 | 75 | 361 | 15 | .711 | 1.108 | 95.5 |
| n-1 | 450 | 1293 | 25 | .095 | 1.276 | 87.0 |
| n-1 | 450 | 1528 | 18 | .115 | 1.466 | 136 |
| p-1 | 230 | 451 | 15 | .592 | 1.369 | 89.0 |
| q-1 | 157 | 908 | 80 | .130 | 1.793 | 76.5 |
| q-1 | 157 | 1089 | 35 | .149 | 1.958 | 205 |

ceeds, therefore, faster for the silica gel than for the sands, indicating that with the gel, less energy is required for the mixing process (the same amount of power, but applied over a shorter period of time). As all three beds were composed of particles of approximately the same composite size, total external surface areas were also approximately equal. If it is assumed that fluidization work done by the gas is dissipated as irreversible frictional work in the bed, it appears that the constant $\beta$ constitutes an inter-particle friction factor. If so, $\beta$ should depend on surface characteristics of the particles and $\beta$ should possibly be related to the angle of repose of the finely divided solids. For the present experimental range and on the basis of the relatively limited accuracy, $\beta$ seems to be comparatively independent on the state of bed expansion, the weight-size distribution of the charge and the composite particle diameter.

The value of fluidization efficiency as a correlation parameter is apparent from

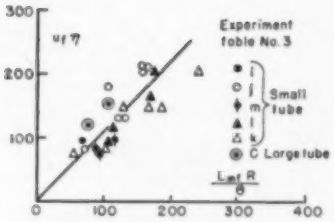


Fig. 4. Silica gel data.

Figures 6 and 7. In Figure 6 particle velocities $L_{mf}R/\theta$ have been plotted against $u_f$, rather than $u_f\eta$, as was done in Figures 2 to 4. If $\eta$ is omitted as a variable, the data do not consolidate into groups. Instead, individual curves, characterized by particle diameter, anchored at characteristic values of $u_{mf}$ on the ordinate are obtained. A more detailed presentation of the data is given in Figure 7, indicating that $\eta$ is a characteristic system variable.

Column heights varied between 3 and 12 in. According to Equation (4), time, $\theta$, required for particles at the top to reach the base of the column should be proportional to the expanded bed height. This has been investigated in detail and was true for comparatively small bed heights (up to 8 in.) in the small-diameter vessel. With higher columns, more time was required than anticipated. Associated with the increase in time was a growing tendency of the charge to form slugs. In beds of 12-in. static height, the time for transporting the particles from top to bottom was roughly three times as long as for beds having a height of 6 in. The 12-in.-high bed of large particles slugged severely, exhibiting virtually piston-like flow of the particles, and the slugging effectively retarded smooth mixing of the dyed particles with the undyed bed portion. Ob-

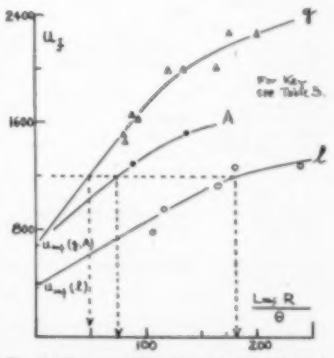


Fig. 5. Turnover data of sands and silica gel particles having approximately equal size.

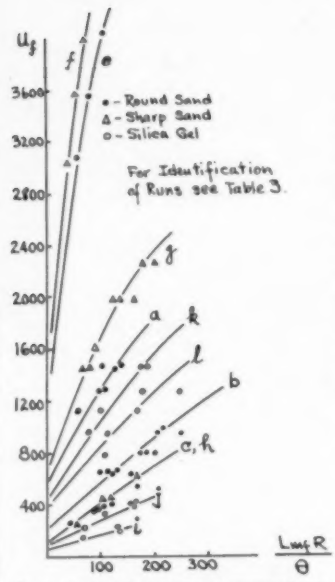


Fig. 6. Turnover data of all beds presented without fluidization efficiency as correlating parameter.

servations suggested that top-to-bottom travel time might be used as a standard for expressing slugging intensity in fluidized charges.

## Heat Transfer

It has been shown in the preceding section how vertical particle velocities along the walls in fluidized columns are related to fluid velocity, extent of bed expansion, height of bed and fluidization efficiency. In the present section a heat-transfer correlation for fluidized beds will be discussed. This correlation was proposed on the basis of a film theory that accounts for the effect upon heat transfer of the vertical velocity of the fluidizing particles along the walls. The apparatus used in the heat-transfer investigation was of the same type as that in which the vertical particle velocities previously discussed were studied. Mass velocity and general particle characteristics were also the same. On the basis of the "particle-film-immersion theory" it has been proposed (5) that:

$$h = 3.0 \times 10^6 k D_p \left( \frac{D_p G_f \eta}{\mu R} \right)^{0.6} \qquad (5)$$

Equation (5) will predict heat-transfer coefficients in B.t.u./(hr.)(° F.)(sq.ft.) if that system is chosen. Since $G_f = u_f \rho$, the relationship between heat transfer and the vertical particle velocity may be shown readily by plotting

$$h/3.0 \times 10^6 k D_p \left( \frac{D_p \rho}{\mu R} \right)^{0.6}$$

against $u_f \rho$. Figure 8 gives such a presentation for a variety of round and sharp sand as well as the iron Fischer-Tropsch catalyst used in earlier studies of heat transfer in fluidized beds (6). The particles differed widely in physical properties and sizes, thus further emphasizing the generality of this treatment of the data. As would be expected from Equation (5) the coefficients increase with the 0.6 power of the vertical particle velocity. Aside from its fundamental value the plot in Figure 8 indicates that heat-transfer coefficients *for this type of equipment* can be estimated if the fluidizing gas velocity and the resulting fluidization efficiency are known. These properties are readily available with a minimum of calculation. The experimental work which led to the definition of fluidization efficiency (7) disclosed that determination of efficiency becomes inaccurate for particles larger than approximately 0.0135 in. For this reason, efficiencies for the two largest sizes of sand particles indicated in Figure 8 were calculated from the *measured* vertical particle velocities

Table 4.- Survey of literature work.

| Authors, reference and operation | Solids | Size range (inch) | Fluids | Pressure range | Linear velocity range, ft./sec. | Type of unit | Vessel diameter |
|---|---|---|---|---|---|---|---|
| Baerg et al (2) (heating) | Iron powder, sands, glass beads, silica, alumina | 0.00462 to 0.03456 | Air | Atmospheric | 0.02 to 2.2h | Internal heating element, bed in annular space. | 1.75 inch in 5.5 inch |
| Campbell and Rumford (3) (cooling) | Sand, Al, Cu powder, graphite, catalysts | 0.0008 to 0.0130 | Air | Atmospheric | 0.15 to 0.35 | Helical coil cooler in vertical position. | 5 inch |
| Leva et al (6) (heating) | Sands, iron Fischer-Tropsch catalyst | 0.00155 to 0.00452 | Air, $CO_2$, He | Atmospheric to 65 p.s.i.a. | 0.05 to 2.0 | Bed inside circular vessel, heat flow through confining wall. | 2 and 4 inches |
| Leva (5) (heating) | Sands, iron Fischer-Tropsch catalyst, silica gel | 0.00632 to 0.0179 | Air, $CO_2$, He | Atmospheric to 80 p.s.i.a. | 0.05 to 2.50 | Same as reference (6) | 2 and 4 inches |
| Lovenspiel and Walton (8) (cooling) | Coal | 0.00519 to 0.0663 | Air | Atmospheric | 0.25 to 5.5 | Same as reference (6) | 4 inches |
| Logwinuk (9) (cooling) | Fused $Al_2O_3$, silicon carbide, silica gel | 0.00345 to 0.0098 | Air, $CO_2$, He | Atmospheric | 0.1 to 0.6 | Same as reference (2) | 3/8 inch tube in 2 inch pipe |
| Mickley and Trilling (10) (heating dilute phase principally) | Glass beads | 0.0016 to 0.0179 | Air | Atmospheric | 1.7 to 8.3 | Same as reference (2) | 0.491 inch Calrod in 2.875 inch tube |
| | | | | | 0.8 to 13.6 | Same as reference (6) | 1 inch |
| Van Heerden et al (11) (cooling) | Carborundum, coke, iron oxide, lead | | Air, $H_2$, $N_2$ | Atmospheric | | Same as reference (6) | 3.4 inches |
| Vreedenberg (12) (cooling) | Sands, iron ore | 0.0092 to 0.0236 | Air | Atmospheric | 0.1 to 0.25 | Large fluidized charge with horizontal or vertical single cooling element. | cooling tube 1.36 inch vessel 22 inches |

Cannon (4), heating; a recorrelation of the data of Mickley and Trilling (10).

Agarwal and Storrow (1), heating; they used a variety of solids in a 1.5 inch diameter tube. Their data are not comparable to the other fluidized bed heat transfer coefficients because they based their coefficients on the logarithmic mean temperature difference as based on terminal conditions, rather than on actual temperature profiles observed in the bed.

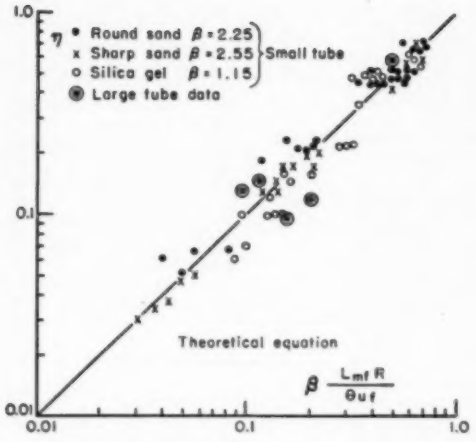


Fig. 7. Data representation in terms of fluidization efficiency. Solid line indicates course Equation (4).

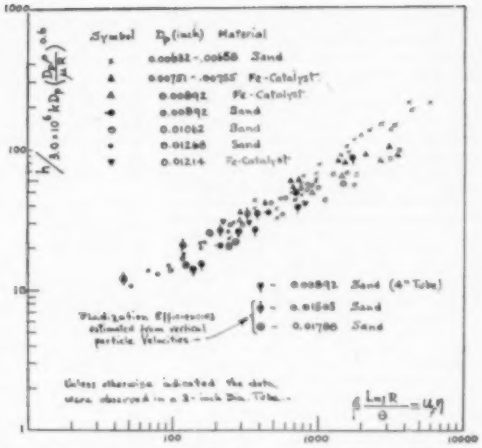
Symbol
Dp (inch)
Material
x 0.00832 - .00858 Sand
▲ 0.00731 - .00755 Fe-Catalyst
△ 0.00892 Fe-Catalyst
◉ 0.00892 Sand
o 0.01062 Sand
• 0.01268 Sand
▼ 0.01214 Fe-Catalyst
ψ - 0.00892 Sand (4" Tube)
Fluidization Efficiencies estimated from vertical particle Velocities -
ϕ - 0.01505 Sand
⊕ - 0.01780 Sand
Unless otherwise indicated the data were observed in a 3" inch Dia. Tube.
h/3.0×10⁻⁵ kDp (Dp G/μR)^0.6
θ Lmf R / θ = (Uf η)
100
1000
10000

Fig. 8. Presentation of heat transfer data in terms of vertical particle velocities along the wall of the vessel.

along the wall, assuming that values of $\beta$ were the same for these sizes of sands as had been found for the smaller sizes ($\beta = 2.55$ and 2.25 for sharp and round sand, respectively). Data, calculated in this way, are in good agreement with all of the other measurements, emphasizing again that heat-transfer coefficients are predictable from a knowledge of particle velocities along the heat-transfer wall.

Experimental data and methods for evaluating fluidization efficiencies for sands, iron Fischer-Tropsch catalysts and coal particles, fluidized in units of the type discussed are available (*7*). The method for evaluation of fluidization efficiency is somewhat involved. Values of $\eta$ have therefore been calculated for particles varying from 0.002- to 0.012-in. diam. These data are plotted in Figure 9 against the ratio $G_f/G_{mf}$, where $G_f$ represents any fluidization mass velocity and $G_{mf}$ the mass velocity required to fluidize the bed initially. The graph is generally valid and may be used with any system of the type discussed here, as long as fluidization is accomplished by a gas. Values of $G_{mf}$ are easily determined, either by experiment or by earlier proposed correlations (*2, 7, 9*).

Previous investigations have revealed that heat-transfer coefficients increase as the particle diameter decreases. This is also true in the present work. It is easily shown that $\eta \propto D_p^{-n}$; where $n$ will vary approximately from 1.8 to 2.2, depending on the flow range considered. If, on the average, $h \propto D_p^{-2}$, it follows from Equation (5) that $h \propto D_p^{-0.4}$, which indicates that the coefficients increase as particle size decreases.

Equation (5) has certain limitations. These are best understood from a general consideration of the present status of fluidized bed heat transfer. The most important details pertaining to the work of others has been summarized here in Table 4. Coefficients of heat transfer have been observed for a considerable range of conditions and correlations of various forms have been proposed. General agreement exists on the following points:

1. Coefficients of heat transfer usually increase considerably when any empty vessel is charged with fluidizable material under the same rates of mass flow.
2. Coefficients increase as particle diameter decreases.
3. Heat-transfer coefficients are practically independent of the thermal conductivity of the solids.

There is less agreement concerning several other important points, notably the effect of density of a fluidized bed on the coefficients and the exact influence which the mass velocity of a gas has in certain operating ranges. However, the most significant deviations are observed when different types of units are used in the investigation. Thus coefficients observed in systems with internal heating or cooling elements are nearly always significantly higher than similar coefficients observed in equipment in which heat flows through the outer confining wall of the vessel.

The study of Baerg and co-workers (*2*) falls into this class and they have pointed out that the particle motion along their centrally located heat-transfer element was much more rapid than the particle velocity

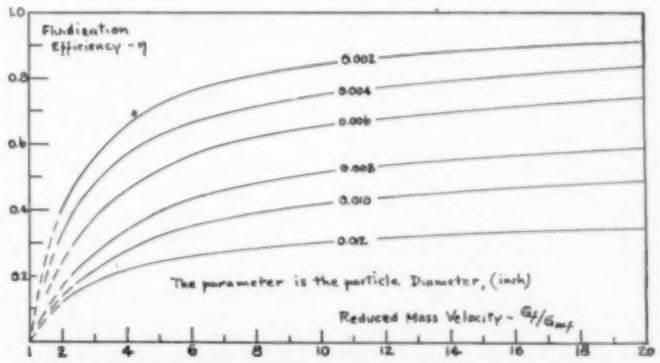


Fig. 9. Fluidization efficiencies shown as function of reduced mass velocity for particles ranging from 0.002 to 0.0120 in.

along the wall. These authors believe that the particle velocity is in general responsible for the improved heat transfer in a fluidized bed. Mickley and Trilling (*10*) have also investigated heat flow with internal as well as external heat-transfer surfaces. Their data do not support this observation as clearly as does a comparison of the data of Baerg and co-workers with the present research. Mickley and Trilling worked, in general, with a much less dense bed and it is conceivable that at low bed densities the difference between internal and external heat transfer is lessened. The few results which they give for relatively dense beds agree with the present work reasonably well. The original observation is supported by the work of Logwinuk (*9*) in which an internal cooling element was used in dense beds. Results of Logwinuk are from 50 to 75% higher than the present data. It is apparent that, for the present, Equation (*5*) must be used only when heat is transferred through the outside wall. Examination of Figure 8 indicates that a vessel diameter variation of from 2 to 4 in. has no effect on heat transfer. Although extrapolation to smaller sizes is probably permissible, it is doubtful whether extension to essentially larger diameters is advisable. With the equipment of relatively small diameter which was employed, horizontal temperature profiles were almost nonexistent. As the vessel diameter increases horizontal temperature gradients will probably be accentuated, with a corresponding effect on the magnitude of the coefficients.

Most previous correlations of data for heat transfer in fluidized beds have been entirely empirical, that is, the effect of fluidization characteristics upon heat transfer have not been considered. An attempt at generalization was recently made by Gamson (*4*). He recorrelated most of the data of Mickley and Trilling and presented them in the form of "*j*-factors," including an empirical bed voidage function. Although dimensional homogeniety was achieved, the correlation of Gamson is hardly more general than the original presentation of Mickley and Trilling.

An interesting treatment of data was presented by Baerg and co-workers (*2*). Using the maximum coefficient already reported by Mickley and Trilling as well as Levenspiel and Walton (*8*) they proposed a correlation in terms of this maximum value and the data observed below the maximum. As already stated they observed the sudden increase in heat transfer as the charge begins to fluidize and made use of this as a new criterion for the onset of fluidization. This constitutes an advance because all previous observations pertaining to the onset of fluidization were purely visual, whereas according to their definition a much more precise determination of the beginning fluidization is possible.

Two interesting papers have recently appeared by Dutch workers (*11, 12*). Van Heerden, Nobel and Van Krevelen (*11*) considered cooling through the outer walls of the apparatus, whereas Vreedenberg (*12*) investigated heat transfer in a large fluidized charge with an immersed horizontal and vertical single cooling element of small diameter. Independently, they related heat-transfer coefficients to the quantity $G_f/G_{mf}$. Van Heerden and co-workers (as well as Logwinuk) termed the value $G_{mf}$ the critical mass velocity (critical as to the beginning of fluidization) and they referred to the ratio $G_f/G_{mf}$ as the "reduced" mass velocity. A correlation of heat-transfer film coefficients in terms of this reduced mass velocity represents a fundamental advance in the solution of the problem similar to that suggested in this paper and in earlier work (*5, 6*). Equation (*5*) does not employ the reduced mass velocities, but instead uses the fluidization efficiency. Figure 9 indicates that for each particle size a direct functional relationship exists between fluidization efficiency and the reduced mass velocity, emphasizing the similarity between the correlations of the Dutch workers and the present work.

If one considers the limits of $G_f/G_{mf}$ and how they pertain to fixed and fluidized beds one finds that for $G_f/G_{mf} < 1$, the bed is fixed, whereas for $G_f/G_{mf} > 1$ the charge will be fluidized. Therefore, the higher the value of $G_f/G_{mf}$ the more violent will be the fluidization action, resulting in higher particle velocities along the heat-transfer surfaces and consequently higher coefficients. Data of Van Heerden and co-workers and Vreedenberg do not agree. Values of Van Heerden are also higher than reported in this paper, whereas the data of Vreedenberg are in somewhat closer agreement with the present work. These deviations are without doubt caused by differences in equipment. Correlations in terms of $G_f/G_{mf}$ and the related fluidization efficiency are therefore even more significant, because they indicate that fundamental heat-transfer correlations in terms of particle velocities *are* possible. It is however clear that before a *universal* heat-transfer correlation in terms of particle velocities can be achieved a better understanding of the dynamics of the fluidized charge and a knowledge of how the pattern of particle motion depends on the unit design will be required.

## Notation

$F$ = force
$M$ = mass
$L$ = length
$\theta$ = time
$T$ = temperature
$H$ = heat
$d$ = differential (no dimension)
$h$ = heat-transfer film coefficient $(H\theta^{-1}L^{-2}T^{-1})$
$k$ = fluid thermal conductivity $(H\theta^{-1}L^{-1}T^{-1})$
$s$ = vertical particle velocity along wall $(L\theta^{-1})$
$u_f$ = linear fluid velocity at fluidization (based on open tower) $(L\theta^{-1})$
$u_{mf}$ = linear fluid velocity at minimum fluidization (based on open tower) $(L\theta^{-1})$
$w$ = weight of bed $(M)$
$A_T$ = cross section of fluidization vessel $(L^2)$
$D_p$ = composite particle diameter $(L)$
$G_f$ = fluidization mass velocity $(M\theta^{-1}L^{-2})$
$G_{mf}$ = minimum fluidization mass velocity $(M\theta^{-1}L^{-2})$
$L_{mf}$ = bed height under minimum fluidization condition $(L)$
$R$ = bed expansion ratio (no dimension)
$\beta$ = inter-bed particle friction factor (no dimension)
$\Delta p$ = pressure drop across fluidized bed $(FL^{-2})$
$\eta$ = fluidization efficiency (no dimension)
$\theta$ = time required for particles to travel from top to bottom of fluidized bed $(\theta)$
$\mu$ = fluid viscosity $(ML^{-1}\theta^{-1})$
$\rho$ = fluid density $(ML^{-3})$

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## Written Discussion

**Arthur D. Holt:** (Jeffrey - Traylor Div., Jeffrey Mfg. Co., Columbus, Ohio): The objective of the study reported in this manuscript is a basic one and sorely needed. The development of an equation and a determination technique for the term, fluidization efficiency, as judged by solids turnover, can be counted a major contribution.

Having had considerable experience in the development, design, and application of industrial equipment for the heat-processing of granular and/or powdered solids that employ for one type a live though not fluidized material bed when transferring heat by the conduction mechanism, and in another type

does employ a fluidized material bed when effecting heat transfer by the contaction mechanism—I should like to state that the authors' basic concepts, their approach to the problem, and conclusions (the latter with one major exception) seem sound. They are supported by their own data which required exacting, painstaking research effort. Ours, while industrial in scope, support their general conclusions (with one major exception). It is pertinent to point out that our equipment is of the continuous-processing type and of course large—whereas theirs was batch-load and unloading. Further, our equipment employed vibration to move the material continuously as well as continually giving it a live and/or fluidized bed condition. This definitely introduces a horizontal-movement component which they assumed to be of small import. That the results in general agree, substantiate their assumption. In short, it is the vertical fluidization action that does the trick.

However, I should like to point out that the employment of a horizontal component, as induced by and obtained by vibratory equipment, enables the obtaining and maintaining a much more uniform distribution of the fluidization action than is possible with beds on stationary equipment or even rabbling equipment. Further, it does so with substantially less pressure drop. Also, though it may seem contrary, it does so while permitting higher gas flow rates before encountering the bugaboo of channeling and slugging.

It is my opinion that while the heat-transfer data have served admirably to support the basic conception of turnover measurement and fluidization efficiency, it will not serve as a basis for extension to substantially larger vessels. For in a large vessel the fluidization pattern would be a goodly number of vortexes or patterns, not just one, as they describe (Fig. 5), thus much material would not contact the walls to permit a heat-pickup.

Hence the heat-transfer data for transfer by the conduction mechanism would not seem extendable and applicable. However, the heat-transfer data from solids to the gas, or vice versa, by the contaction mechanism in a fluidized bed would be extendable and applicable for equivalent conditions, though they will be most difficult of obtainment. Also the values will be much higher (some three to five times with 10 times not unusual) than for the conduction-mechanism phase. They expressed some doubt about this. Our experience would also indicate that they can go much further in larger particle-size treatment than they apparently anticipate, as we regularly fluidize for heat transfer—material 20 mesh to 3/16 in. in size, as well as smaller, and as well as mixed, and generally with a one-minute or less exposure. If drying is also involved or is the major objective—such with its greatly increased heat load takes longer, one to three minutes being a general practice figure.

*(Presented at A.I.Ch.E. Forty-third Annual Meeting, Columbus, Ohio.)*

# ABSTRACTS

EDITOR'S NOTE: A continuation from the May issue of the abstracts of papers published in "Phase-Equilibria—Pittsburgh and Houston," Chemical Engineering Progress Symposium Series No. 2, Vol. 48 (1952). The volume may be ordered from Chemical Engineering Progress, 120 East 41 Street, New York 17, N. Y.*

## PHASE-EQUILIBRIA IN THE SYSTEM PROPYLENE OXIDE-WATER

*J. N. Wickert, W. S. Tamplin, and R. L. Shank*

Carbide and Carbon Chemicals Corporation

THE phase-equilibrium relationships of the system propylene oxide-water from the vapor state to the solid state were investigated and the data evaluated by calculation of the activity coefficients or eccentricity factors. The relations were investigated at atmospheric pressure, the experimental data being corrected to 760 mm. of mercury absolute pressure, and the system was found to form two liquid phases over a certain composition range at the atmospheric boiling point and yet not to form an azeotrope. This behavior can occur only with partially miscible liquids of greatly different volatilities.

*Chem. Eng. Progress Symposium Series*—"Phase-Equilibria--Pittsburgh and Houston," **48**, No. 2, 92 (1952).

## PREDICTION OF HYDROCARBON VAPOR-LIQUID EQUILIBRIA

*Elliot I. Organick and Geo. Granger Brown*

University of Michigan

THIS paper develops a correlation for predicting equilibrium-volatility ratios, $K = y/x$, for hydrocarbon systems, based on experimental data for binary systems on normal paraffins containing methane and a convergence-pressure variable. By employing an empirical relationship based on critical pressures for methane binary systems, the convergence pressure is defined in terms of the equilibrium pressure, the molal average boiling point of the vapor, and the weight-average-equivalent-molecular weight of the liquid. The equivalent molecular weight is also used to compute the "equivalent mole fractions," $y$ and $x$, which define the value of $K$. Published binary and complex experimental vapor-liquid-equilibrium data for hydrocarbons agree with values indicated by the correlation in all ranges of temperature and pressure within the experimental uncertainty.

*Chem. Eng. Progress Symposium Series*—"Phase-Equilibria--Pittsburgh and Houston," **48**, No. 2, 97 (1952).

## EQUILIBRIUM VAPOR AND LIQUID FROM FLASH VAPORIZATION OF PETROLEUM FRACTIONS

*Wayne C. Edmister and John R. Bowman*

Carnegie Institute of Technology and Mellon Institute

THE true-boiling-point-distillation curves of most petroleum fractions may be represented by straight lines on a log $\alpha$ ($\alpha$ = relative volatility) vs. fraction distilled plot. For this case a method is presented for computing the T.B.P. curves of the equilibrium vapor and liquid products from flash vaporization. Charts for applying this method and an illustration are presented. The charts include a graph of $\alpha$ vs. temperature for lines of constant boiling point and plots relating points on product T.B.P.'s with the corresponding point on the feed T.B.P.

*Chem. Eng. Progress Symposium Series*—"Phase-Equilibria--Pittsburgh and Houston," **48**, No. 2, 112 (1952).

## SIMPLIFIED NOMOGRAPHIC PRESENTATION—Hydrocarbon Vapor-liquid Equilibria

*Francis W. Winn*

Socony-Vacuum Laboratories

A CORRELATION of the vapor-liquid equilibrium $K$ ratio of the light hydrocarbons, narrow-cut petroleum fractions, and certain nonhydrocarbon systems is presented in nomographic form as a function of the temperature and pressure of the system and the composition of the liquid phase. The correlation consists of a nomogram that may be applied to nonazeotropic systems of the paraffin hydrocarbons from methane through heptane and of the olefins and aromatics of corresponding boiling points and to certain nonhydrocarbon systems; a coordinate plot with which the nomogram can be used; and another coordinate plot and an equation which are used in conjunction with the nomogram to obtain the $K$ ratios of high-boiling pure compounds and narrow-cut petroleum fractions.

*Chem. Eng. Progress Symposium Series*—"Phase-Equilibria--Pittsburgh and Houston," **48**, No. 2, 121 (1952).

* *See advertisement, page 64, News Section, for details.*

# ENTHALPY-CONCENTRATION CHART

## FOR THE SYSTEM $H_2O$-$SO_3$

WILLIAM D. ROSS

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THE enthalpy-concentration chart has been discussed by McCabe (*4*) who presented such a chart for $H_2SO_4$ based on data plotted by Zeisberg (*7*). Hougen and Watson (*2*) presented a similar chart. Both charts are limited to a maximum strength of 100% $H_2SO_4$, and neither is drawn to permit reading temperatures as closely as 2° F. (*8, 9*).† The first such chart which was drawn for use here resembled these in using wt. % $H_2SO_4$ as abscissae. This proved unsatisfactory, because the portion of the chart for concentrations above 75% was unduly compressed. The present chart (Fig. 1) uses mole % $SO_3$ as abscissae. This results in proportions more suitable for use. The scale of wt. % $H_2SO_4$ is added for convenience.

Mole fraction $SO_3$ is defined as

$$x = \frac{\frac{P}{98.08}}{\frac{2P}{98.08} + \frac{(100-P)}{18.02}} \qquad (1)$$

where

$$P = \text{wt. \% } H_2SO_4$$

98.08 and 18.02 are the molecular weights of $H_2SO_4$ and of $H_2O$, respectively.

The ordinates are given in terms of kilogram calorie per gram mole (which is the same as thousands of P.c.u. per pound mole), where one gram mole of solution is defined as

$$80.06x + 18.02(1-x)\text{g.} \qquad (2)$$

In computing the curves, 16° C. was chosen as the base temperature, and, to obtain curves of the most satisfactory shape over the range of compositions covered, the standard states were chosen as

$H_2O$ = 0 kg.-cal./g. mole and
100% $H_2SO_4$ = −1.70 kg.-cal./g. mole

If a chart were drawn to cover either a narrower or a wider range of compositions, other definitions of standard states would be more suitable.

Data used in plotting the 16° C. curve were heats of dilution compiled by Dee (*1*) and by Morgen (*5*). Data of Dee's Tables I, II, and III, and of Morgen's Table I, were separately recomputed to fit the standard states as previously defined, and the points plotted. The data were in fairly good agreement in the region of dilute acid, but there was enough scattering in the region of concentrated acid to amount to an error in temperature of several degrees. In this region, the curve was determined from earlier measurements of the temperature rise in mixing 77.5% $H_2SO_4$ and 104.5% $H_2SO_4$ to various strengths.*

Specific heats used between 0 and 100% $H_2SO_4$ were those reported by Sokolik (*6*), for 20°, 40°, 60°, and 80° C., and extrapolations to higher temperatures. The specific heats of oleum were taken from Knietsch (*3*); the variation with temperature was assumed to be the same as with 100% $H_2SO_4$.

The calculated curves are found consistent with the temperature rises in mixing acids of various strengths, measured by various workers in Du Pont laboratories.

### Use of Chart

When two portions of acid at any two concentrations and temperatures are mixed, the temperature of any mixture will lie on the straight line joining the points corresponding to the initial acids.

**Problem 1.** Mix 65.0% $H_2SO_4$ at 24° C. and 104.0% $H_2SO_4$ at 16° C. to give 79.0% $H_2SO_4$.

(a) What will be the temperature of the mixture?

(b) How much heat must be removed from the mixture to cool it to 30° C.?

**Solution.** (a) Draw a straight line joining the points representing the two original acid concentrations. The temperature at 79.0% $H_2SO_4$ is read as 90° C.

(b) The enthalpy at this point is −2.20 kg.-cal./g. mole of solution. The enthalpy at 79.0% $H_2SO_4$ and 30° C. is read as −3.21 kg.-cal./g. mole. The heat which must be removed is therefore

$$3.21 - 2.20 = 1.01 \text{ kg.-cal./g. mole of solution or } 1010 \text{ P.c.u./lb. mole}$$

From the chart, for 79.0% $H_2SO_4$, $x = .291$. Therefore by Equation (*2*), one pound mole is

$$80.06 \times .291 + 18.02 \times .709 = 36.1 \text{ lb.}$$
$$1010 \text{ P.c.u.} \div 36.1 \text{ lb.} = 28.0 \text{ P.c.u./lb.}$$

From the chart it is possible to determine the proportions in which three components must be mixed to give a final mixture of specified concentration and temperature.

**Problem 2.** We have 65.0% $H_2SO_4$ at 24° C., 104.0% $H_2SO_4$ at 16° C., and 20% $H_2SO_4$ at 12° C.

(a) What quantities of each must be used to give 100 lb. of 70.0% $H_2SO_4$ at 100° C.?

**Solution.** Draw the straight line joining the points representing the 65% and the 104% acids. Draw another straight line between the points representing the 20% acid at 12° C. and the 70% acid at 100° C., and extend it until it intersects the first line. The point of intersection is read as 78.7% $H_2SO_4$. This means that the first two acids will be used in proportions such as to give 78.7%, and enough 20% acid added to dilute this to 70%. The quantities required are calculated by material balances:

Total weight of all acid = 100 lb.

$H_2SO_4$ balance:

$$\text{lb. of 20\% acid} \times 20 + (100 - \text{lb. of 20\% acid}) \times .787 = 100 \times .70$$

$$\text{lb. of 20\% acid} = \frac{100(.787 - .700)}{.787 - .200} = 14.8$$

$$\text{lb. of 104.0\% acid} + \text{lb. of 65.0\% acid} = 100 - 14.8 = 85.2$$

$$\text{lb. of 104.0\% acid} \times 1.040 + (85.2 - \text{lb. of 104.0\% acid}) \times .650 = 85.2 \times .787$$

$$\text{lb. of 104.0\% acid} = \frac{85.2 \times (.787 - .650)}{1.040 - .650} = 29.9$$

$$\text{lb. of 65.0\% acid} = 85.2 - 29.9 = 55.3$$

For problems involving steam, the vertical axis at 0% acid can be extended. The ordinate for saturated steam at 100° C. is 11.22 kg.-cal./g. mole. This can be measured as 25.1 cm. (9⅞ in.) above the 2.0 kg.-cal. ordinate.

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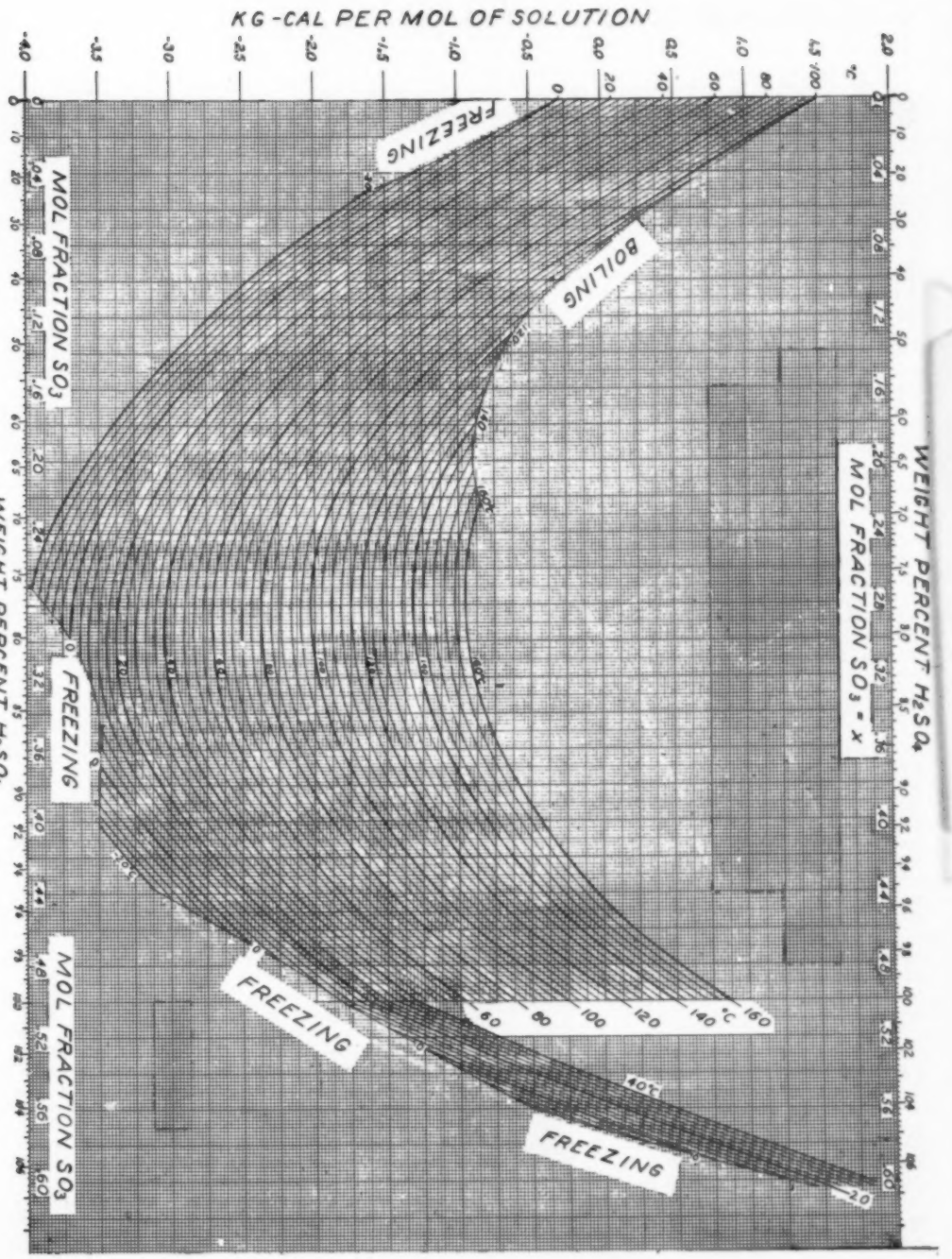
† See footnote col. 1.

† Diagrams using as abscissae weight % $H_2SO_4$ in the range 0-100% and % free $SO_3$ for oleum have been published (*8, 9*). These also are not suitable for all the purposes of this paper.

* Measurements by E. N. Kramer, of the Du Pont Co.


KG-CAL PER MOL OF SOLUTION
FREEZING
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MOL FRACTION SO3
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FREEZING
FREEZING
FREEZING
MOL FRACTION SO3
WEIGHT PERCENT H2SO4

Fig. 1.

# Collective Case Histories of

# STUDENT CONTEST WINNERS

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LAST year the editor of "C.E.P." wrote the chairman of the Student Chapter Committee of A.I.Ch.E. that an article dealing with the employment history and present occupation of the winners of the A.I.Ch.E. Student Contest Problem since its inception would be interesting. This problem originated in 1932, and since then, with the exception of the war years 1944-46 inclusive, the award has been made annually. The present prizes are $200 for first prize, $100 for second prize and $50 for third prize, with three honorable mention Award Certificates being given in addition to the cash prizes, making a total of six awards annually. In two of the years between 1933 and 1950 the Judging Committee did not award all six honors so that the total number of student prize winners for that period was 79.

**Membership in A.I.Ch.E.** Questionnaires were sent to all award recipients whose addresses could be found, and the data presented here were obtained from the information received in the responses. From the seventy-nine quizzed, sixty responses were received. Nineteen individuals could not be reached either because the addresses used for them were not correct, or because no address could be found. Repeated attempts were made to locate these men.

The author, having been a member of the A.I.Ch.E. Student Chapter Committee for several years, along with many other members of the A.I.Ch.E., has been concerned about the fact that only about 10 per cent of the seniors graduating in chemical engineering in the United States and Canada join the A.I.Ch.E. as Junior members immediately upon their graduation. In this investigation it was found that twenty-eight of the seventy-nine winners, or about 35 per cent are now members of the A.I.Ch.E. as shown by the 1950 Year Book. Out of the 60 respondents all but one are either practicing chemical engineering or are in jobs, oftentimes of a supervisory nature, as a direct result of having practiced chemical engineering.

The Student Membership grade initiated two years ago seems to be improving the percentage of graduating students who join A.I.Ch.E., but the Student Membership grade has not been in existence long enough to allow prediction of its ultimate result.

Little information in the responses permitted conclusions on the reasons for the failure of most of the winners to become members of A.I.Ch.E.; the only statement in any of the responses pertaining to this question was the following, "---oddly enough I never joined the A.I.Ch.E. after leaving school because of ineligibility as a result of the combination of its requirements on age limits and years of experience."

**Occupation.** Table 1 indicates the work-distribution pattern, past and present, of those sixty from whom replies were received. It is shown that process engineering comprises the largest single type of present occupation followed by research and development, manufacturing and production work, and then by executive positions. Table 1 also shows that there is no one type of job which leads predominantly to executive positions, because those men now employed as executives have experience records equally divided between the three other principal types of experience. In interpreting Table 1, it will be obvious that men now employed as executives formerly worked in a variety of jobs. The same is true of men now employed in such areas as process or in research and development, and in most cases these men have moved about among several different jobs before reaching their present positions. Hence, the number of jobs held in the past exceeds the number of men involved by a good margin.

In constructing Tables 1 and 2, many arbitrary decisions had to be made. For example, there was a question as to when a man could be considered an executive just as there was one as to when a man could be considered an employee in process engineering rather than in research work. In these doubtful cases the author used his best judgment in classifying the type of job now held. In general, the arbitrary decision was made that a man would not be considered an executive unless he was the head of a reasonably large organization. A group leader in a research organization was not classified as an executive, but rather was considered as being in research, even though he might have had ten or twelve men under his immediate supervision. To fall in the executive classification then, the man would have to be a director of research, a manager of operations, or a vice-president. The same general arbitrary decisions were necessary in classifying other job types. Where it was not known clearly from the questionnaire whether a man could be classed as being, for example, in research rather than in process engineering, a decision was reached based upon his job title, the

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author's knowledge of the company's type of operation, or the general tenor of the man's letter written in answer to the inquiry. These uncertainties arose mostly in the case of the older men because new graduates generally were easy to classify.

**Effect of Graduate Degree Obtained on Occupation.** Table 2 is an attempt to show the type of job now occupied on the basis of the ultimate degree obtained. Because of the small numbers involved and the corresponding uncertain statistical conclusions to be expected from sixty case histories, it is not clearly indicated that the Ph.D., or Sc.D., or M.S. leads to executive jobs to any greater extent than does the B.S. degree. As shown by Table 2, seven out of sixty are in executive positions, and four of these executives have completed graduate work leading to the M.S., Ch.E., or Ph.D. degree while the three B.S. men have had no graduate training. Within the accuracy of the data, however, it is indicated that graduate training helps make an executive.

Table 2 shows quite plainly that men with advanced degrees tend to go into research and development to a greater extent than do the B.S. men. One third of the advanced degree men are in research, while only about 13 per cent of the B.S. men are so employed. Of the Bachelor men 72 per cent are engaged in process work, while only 17 per cent of the men with advanced degrees are engaged in such work. Of the B.S., 20 per cent, and 26 per cent of the M.S. men are in manufacturing.

Thirty-four of the sixty winners took graduate work resulting in eleven Ph.D.'s (most of whom would also have M.S. degrees), two Ch.E.'s and seventeen M.S. degrees. Four men enrolled in graduate work but did not get an advanced degree. Only two of the contest group are now professors, and only one other man at some time did college or university teaching at the professorial level. A number of other men reported teaching as instructor or lower level while in graduate school. A number of men secured part or all their graduate work in night school. Judging from the low percentage of winners who have chosen teaching as their career, teaching is not an attractive field.

It may be thought surprising that out of the twelve men now engaged in manufacturing, nine have had graduate instruction—a higher percentage than in any other occupations except teaching and research. Six advanced degrees resulted from this graduate work. This shows that three of the four men who dropped graduate work without obtaining an advanced degree are now in manufacturing. In examining Table 1, it should be noted, for example, that if a man is working now as a process engineer, his experience record will not show previous experience as a process engineer, so that in all cases there will be fewer numbers in any single "other experience" column than in the "present occupation" column.

**Salaries.** It was thought desirable to get an indication of the present salaries received by the prize winners, even though there is no satisfactory way of comparing these salaries with those received by chemical engineering graduates as a whole. Figure 1 shows the average present salaries for those who answered this part of the questionnaire.

Individual points are not shown in Figure 1 because several individuals requested that the information not be itemized in detail. Accordingly, the data were plotted and the best line drawn through the points to show averages. A few individual points deviated from these curves by as much as 50-75 per cent or even more, above the curve in the higher salary brackets, but in spite of these individual large variations, averages defined the curve with little deviation. There were insufficient data points to enable medians to be used satisfactorily.

Earnings of the Ph.D. men show a clear superiority over Bachelors and M.S. men, but assuming (as is indicated in Figure 1) a period of three years between the Bachelors and the Ph.D. degrees, the data indicate that the Ph.D. degree, compared with the M.S. and B.S. degrees, does not show a profit until after the eleventh year, at the end of which period B.S. and Ph.D. men will have earned about $55,400 each.

This may be misleading if one takes into account the income tax picture of the future, where the higher earnings of the Ph.D. men may be largely lopped off by surtax rates. An estimate of the effect of present and past tax rates, however, indicates that the picture has not changed much with taxes, and that it would have taken a little more than eleven years for the Ph.D. man to catch up in net earnings after Federal taxes. This is in close agreement with the result without taxes; the close agreement being caused by the fact that men without Ph.D. degrees pay taxes for three years while the Ph.D. man is attending school and presumably paying no taxes. In making these calculations it was assumed that the Ph.D. man would be largely self-supporting through these three years. If he were to borrow money to attend school, and the debt subtracted from earnings the pay-out time for the Ph.D. would be longer. The assumption is believed justified because with the present shortages of students, most Ph.D. candidates can avail themselves of fellowships and grants and be self-supporting.

The returns showed that those with M.S. degrees started out at a slightly higher salary than the B.S. men, but after about three years there was no perceptible difference between the earnings of the M.S. and the B.S. men. During later years more Bachelors deviated above the curve than did M.S. men. A survey made for the A.C.S. in 1944 indicated the same conclusion (*1*).

With a pay-out time of about eleven years for the Ph.D., compared to the B.S., and with the likelihood of higher surtax rates lengthening the pay-out time, industry and education will find it necessary to devise added incentives to make graduate work attractive.

Other interesting points, not discernible from Figure 1 because individual points are not shown, are:

a. Engineers employed during the period 1948-50 at the B.S. level sometimes were paid higher starting salaries than engineers, hired three or four years previously, were earning in 1948-50. This is not true in the majority of cases, otherwise the curve would not show a consistent positive

TABLE 1

| PRESENT OCCUPATION | PRESENT NUMBER OF MEN SO EMPLOYED | OTHER EXPERIENCE: MILITARY SERVICE | MANUFACTURING | PROCESS | SENIOR PATENT ATTORNEY | PROFESSOR | RESEARCH & DEVELOPMENT | SALES | SUPERVISORY -EXECUTIVE | GRADUATE STUDY |
|---|---|---|---|---|---|---|---|---|---|---|
| Military Service | 1 | | 1 | | | | | | | |
| Manufacturing | 12 | 2 | | 5 | | | 6 | 1 | | 9 |
| Process | 10 | | 7 | | | | 9 | 3 | | 8 |
| Senior Patent Attorney | 1 | | 1 | 1 | | | 1 | | | 1 |
| Professor | 2 | 1 | 1 | 2 | | | 2 | | | 2 |
| Research and Development | 14 | 3 | 3 | 0 | | | | | | 10 |
| Sales | 3 | | 1 | 2 | | | | | | |
| Supervisory - Executive | 7 | | 5 | 5 | | 1 | 5 | | | 3 |
| Graduate Study | 3 | | | | | | | | | |
| Diplomatic Service | 1 | | 1 | 1 | | | | | | 1 |
| TOTAL | 60 | 6 | 20 | 23 | 0 | 1 | 23 | 4 | 0 | 34 |

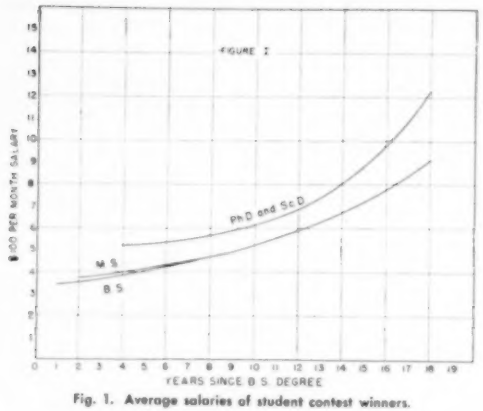



Fig. 1. Average salaries of student contest winners.

TABLE 2

| | PRESENT OCCUPATION | PRESENT PRODUCT TECHNICAL WORK AND | |
|---|---|---|---|
| | | Dev. Dr. | Prod. or Sales |
| Military Service | 1 | 1 | |
| Manufacturing | 12 | 6 | 5 |
| Process | 13 | 13 | 3 |
| Patent Work | 1 | | 1 * |
| Professor | 2 | | 2 |
| Research & Development | 14 | 4 | 6 |
| Sales | 1 | 1 | |
| Supervisory - Executive | 7 | 3 | 2 |
| Graduate Study | 3 | 1 | 0 |
| Diplomatic Service | 1 | 1 | |
| TOTAL | 60 | 30 | 27 ** |

* Also Law Degree

** 17 B.S., 2 Ch.E.

slope, but there were enough individual points above the curve to substantiate this statement. The slow rate of rise of the curves in the early years reflects this condition.

b. Those men employed during the depression period 1933-36 inclusive, are in a number of instances, still suffering from the low salaries paid during that time. Stating this in another way, those men who started out at a low salary in the depression years, in a few cases, have not been increased rapidly enough as they gained experience to bring them up to the level of men employed more recently.
c. From the salary viewpoint, 1938 and 1941 were good years to begin working.
d. Individuals, after a few years' experience, deviate above and below the curve to a considerable extent at all educational levels.

**Geographical Distribution.** Table 3 is an attempt to show present geographical residences of the winners compared with the school from which they graduated. The geographical locations were designated arbitrarily as East, Middle West, West, and Texas. Originally it was thought that a high percentage of winners might have migrated to Texas because of the large industrial developments started there at approximately the same time as the first annual contest. When it was found that this mass migration had not occurred, the orginal arbitrary geographical division was retained to please Texans who might read this paper. The boundary line between East and Midwest was chosen as between Ontario and Manitoba, Indiana-Illinois and then south following the Ohio and Mississippi Rivers. Between West and Midwest the line was chosen along the Dakota-Montana and Oklahoma-Colorado borders and thence south to the Texas border.

Out of the seventy-nine winners, thirty-eight came from the East, twenty-four from the Midwest, twelve from the West, and five from Texas. The present residences of the sixty winners who answered the inquiry showed that there were forty living in the East, five in the Midwest, eight in the West, four in Texas, one in Canada, one in The Netherlands, and one in Germany. Two winners were from Canada, and one remains there, the other having moved to the United States.

This shows plainly that there has been a distinct net movement eastward, and that Texas has just about held its own, while the Midwestern winners have tended to move eastward to a somewhat greater extent than have the far westerners. This comparison may not be due solely to attractiveness of industrial jobs in the East, because many of the winners attended graduate school in the East even though they may have obtained the B.S. degree elsewhere. There has been an extensive amount of trading locations. For example, while there were no winners from the State of California, two have moved there from Oregon, and one each from Illinois, Washington, New York, and Colorado, making a total of six contest winners now residing in California. Only four out of the sixty moved out of the New York-New Jersey area, while seven moved in. All the Texans who responded to the questionnaire had stayed in Texas, and one had moved in from an adjoining state. Otherwise there was no marked trend as to geographical location with the exception that the states bordering on the Gulf of Mexico (other than Texas) are low in the number of winners who live there. Out of seven winners from Oregon six have moved out of the state. The trend toward industrialization both in the deep South and in the Northwest may soon reverse the present scarcity of engineers in these two localities.

## Recommendations

In the questionnaire sent to the winners it was suggested that they offer suggestions for: (a) students, (b) industrial concerns, and (c) educational institutions. Numerous such suggestions were received. There was little agreement among the men making the suggestions. In fact, there were only two cases where as many as four men made the same or similar suggestions. These two cases were:

1. Students should cultivate ability to write clear, concise reports, grammatically as well as technically correct. In many cases it is the best, if not the only, method of contact with their superiors.
2. ---attend evening schools for graduate study whenever possible.

Because some of the remaining suggestions are novel it has been thought worth while to include part of them. These are:

Every young man strives for an executive position, but it is more fun down under.

Students should, as early as possible, gain some knowledge of the broad and varied fields for chemical engineers and guide

TABLE 3

GEOGRAPHICAL LOCATION OF WINNERS

| | EAST | | MIDWEST | | TEXAS | | WEST | | MISC. | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Number | % | Number | % | Number | % | Number | % | Number | % |
| The 79 winners obtained their B.S. Degrees in | 38 | 48.1 | 24 | 30.4 | 5 | 6.3 | 12 | 15.2 | 0 | 0 |
| The 60 winners responding to questionnaire now live in | 40 | 66.7 | 5 | 8.3 | 4 | 6.7 | 8 | 13.3 | 3 | 5.0 |

themselves into teaching, design, sales production, research, etc., and not leave this to chance.

Students should endeavor to take a course, if such a course could be given, in human relations so as to get along with their boss, colleagues, and those people under their supervision. There is a good booklet along this line entitled "12 Pointers that Lead to Promotion" by Richard H. Moulton available from the Updegraff Press, Ltd., Scarsdale, N. Y., for 30 cents per copy.

It is recommended that all young chemical engineers apply for professional engineers' licenses as soon as they can after beginning their industrial career.

Take courses in public speaking and salesmanship (unless you sell you starve).

Many students starting out in industry fail to see or remember the limitations that are necessarily involved in using equations. Although the men were anxious to attack a problem, they generally were amazed to learn that such equations or principles just cannot be applied without discretion to all industrial processes. Every student should systematically read and catalog information found in the technical journals while still in college. This system is encouraged by many employers.

Do not hesitate to take on a menial job. Seldom do graduates step right out of college into a company in the capacity for which he was trained.

At least once or twice a year have a private chat with your boss and learn your weak and strong points. Then prepare a plan or budget for future expenditure of your time, money, and effort on projects beneficial to your growth in industry and important to you and your family.

Learn to type. It saves time in writing notes and reports in rough draft, and is much more easily read.

Begin as early as possible to build a library.

One of their major unsolved problems is that of personnel relations. Much more effort should be given to selecting the proper man for the job and the proper job for the man.

It is recommended that industrial organizations institute a limited training program for new engineers. Such a program would be beneficial to both the engineer and the organization.

To industrie' companies,---I would reiterate a fact which is well known to all well-managed industrial organizations—inspiring leadership is essential in handling all personnel, including the technical people. A good chemical engineer with high potential capabilities will work neither long nor well for inadequate, uninspiring supervision.

Engineering should be a five-year course, emphasizing business law, economics, psychology, and patents.

Colleges should stick to fundamentals and not put much emphasis on specialty courses. The latter can be learned better and faster in industry, but fundamentals not learned in college will probably not be learned at all.

Unit Operations laboratory gives the engineer his first grasp of an individual problem whose solution lies in his personal efforts and originality.

The demand is for *good* chemical engineers. Educational institutions should not lower their academic standards.

Encourage students to give thought at an early stage in their career as to whether they will want ultimately to be attached to a large company, or to a smaller one, or possibly their own. Conscious selection of the latter choice might lead to a job selection more consistent with the desired end result.

It is suggested that at least one course should emphasize practical procedures and experimental techniques in research and development work. Most graduates of the chemical engineering schools can handle the literature search and theoretical computations required for a particular project, but without extensive training few are able to organize an experimental program and visualize the practical experimental setup required to demonstrate the desired process.

Teachers should caution young graduates against the error of 'looking down their noses' at employment in drafting rooms of engineering or operating companies. A year or so on the boards is excellent background training for a young chemical engineer and will stand him in good stead in later years.

Educational institutions could do a lot more toward teaching students the realities of chemical engineering practice.

## General

Other information obtained from the responses has been thought interesting enough to include here.

The diversity of jobs and types of projects in which the men are or have been engaged, ranges from (a) research on and construction of an artificial kidney, (b) early development work and placing in commercial production of streptomycin, (c) wage control, and (d) diplomatic service. One man now occupies a responsible position in the U. S. Foreign Diplomatic Service. One of the men, while in military service, witnessed the surrender ceremonies on the U.S.S. Missouri. One of the contest winners won the William H. Walker Award.

A number of the winners are active in A.I.Ch.E. affairs, being members of various committees such as the Admissions Committee, Education Projects Committee, and the Student Chapters Committee. One is an assistant editor of the A.I.Ch.E. Student Chapter News. Two of the winners helped prepare later student contest problems after they had begun working in industry.

In addition to the suggestions which were made and which have been presented in an earlier section of this paper, there were a few general comments; some were thoughtful and pertinent and some were amusing because they were made in a whimsical manner.

One of the winners who occupies a highly responsible position in the patents field states, while he never gave a moment's thought to patent work when he was in school, he has since found the field to be fascinating and instructive because patent work is a combination of engineering and law, and because the patent literature is "always years ahead of textbooks and often even of the journals. In many cases it contains the only details ever published—"; colleges should therefore emphasize in some portion of their curricula the study of patents and patent literature, he claimed.

Another recent contest winner brings out the fact that once a company, perhaps after an interview, makes a job offer to a student the company expects a prompt decision. This is not at all unreasonable on the part of the company. On the other hand, the student may wish to interview representatives of other companies who will be on the campus at a later date and, therefore, the student is reluctant to reach a prompt decision. The suggestion was made that companies who regularly carry out interviews should reach a gentleman's agreement to make their offers simultaneously at some agreed-upon definite time.

The lack of good safety policies in colleges and universities was criticized by one of the winners who stated that men would be "fired from industry for practices used in schools." Another man liked business administration courses and mentioned specifically those at M.I.T. and Harvard as being valuable. Another felt that theses should be required for B.S. men because these men were expected to have originality after they became employed and in school the thesis-type work was about the only way they could display technical originality prior to leaving school.

Among the more diverting comments were the following: "---I promptly married the girl who typed my student contest problem---," and another, "---I was satisfied to start in the yard gang. It was not many weeks before I was transferred to the control laboratory, and I never did determine whether this was, (1) due to the need for extra help in the laboratory, (2) part of their plan for young technical men, or (3) because they realized I would not last long doing such strenuous manual labor---after four years of academic life---." Another one stated that he obtained an assistant instructorship in chemical engineering because "I think [the] professor had some equipment he wanted fixed---." Another winner stated "---at this point I decided that practical experience would do more good than continuing on for a Ph.D. degree, and also would pay for groceries." Another busy man stated, "I finished finals June 2, got married June 4, graduated June 12, and started graduate school . . . June 16. The dates are approximate because things happened so fast." Another anxious young man went to assume his new job following graduation "---with a lump in my throat and 'Groggins' under my arm I arrived---." Another man said, "---I won the Bronze Medal, not for gallantry, but for long hours of paper work."

## Literature Cited

1. Fraser, Andrew, Jr., *Chem. Eng. News*, 22, No. 16, 2112 (1944).

# CAST ALLOY REFERENCE SHEET

N. S. MOTT, Chief Chemist Metallurgist

The Cooper Alloy Foundry Co., Hillside, N. J.

**ALLOY: CORROSION RESISTANT CAST NICKEL.**

**APPLICATIONS AND REMARKS:** Nickel is mainly used for food handling, although it is absolutely necessary for molten caustic, and has the best resistance to anhydrous halogens and halogen acids at elevated temperatures. It has found some use in handling dilute reducing acids in the absence of air or oxidizing agents. Nickel is cast into valves, fittings, and miscellaneous shapes for these applications. Lower strength and hardness, together with the large increase in cost of 42 cents a pound over 18-8S, make its use somewhat limited to cases of necessity.

**DESIGNATIONS:** CAST NICKEL.

**CHEMICAL COMPOSITION RANGE:** C .2-.7%; Ni > 97%; Si 1-1.5%; Fe < 2%.

**MACHINABILITY:** Commercial nickel, although quite soft, machines fairly well. It contains enough impurities such as sulfur, lead, and traces of low melting alloys to be slightly brittle and chips break up readily on machining. A fine cut and great care must be taken to avoid a granular finish.

**HEAT TREATMENT:** Not heat treated—used in "as-cast" condition.

**WELDABILITY:** Nickel may be welded by the usual methods except the atomic hydrogen, since in the molten state it has a great affinity for gases which are absorbed when in the molten state and released upon cooling, causing gas porosity. Excessive sulfur or lead embrittle the casting and will cause cracks adjacent to the weld.

## MECHANICAL AND PHYSICAL PROPERTIES:

| | | | |
|---|---|---|---|
| Tensile Strength, 1000 lb./sq.in. | 55 | Melting Point | 2540-2600° F. |
| Yield Point, 1000 lb./sq.in. | 25 | Specific Heat (B.t.u./(lb.)/° F.) 32-212° F. | 0.13 |
| Elongation, % | 20 | Thermal Expansion (× 10⁻⁶ in./(in.)/° F.) 32-212° F. | 7.4 |
| Reduction in Area, % | 22 | Thermal Conductivity (B.t.u./sq.ft./° F./in.) 32-212° F. | 410 |
| Brinell Hardness | 100 | Electrical Resistance (Ohms/cir. mil. ft.) @ 32° F. | 125 |
| Charpy Impact (Std. Keyhole ft.lbs.) | 65 | | |
| Mod. of Elasticity (× 10⁶ lb./sq.in.) | 21.5 | | |
| Specific Gravity | 8.34 | | |

## CORROSION RESISTANCE

| | |
|---|---|
| **ACIDS** | |
| Acetic 5%, 70 F | E |
| Acetic 5%, boiling | G |
| Acetic 80%, 70 F | G |
| Acetic 80%, boiling | G |
| Acetic Glacial, 70 F | G |
| Acetic Glacial, boiling | G |
| Benzoic 5%, 70 F | G |
| Boric 5%, 176 F | G |
| Chromic 10%, 70 F | G |
| Chromic 10%, boiling | N |
| Chromic 50%, boiling | E |
| Citric 5%, 70 F | G |
| Citric 25%, boiling | G |
| Citric 50%, boiling | G |
| Formic 5%, 70 F | G |
| Hydrochloric 1%, 70 F | G |
| Hydrochloric 1%, boiling | N |
| Hydrochloric, 5%, 70 F | F |
| Hydrochloric 5%, boiling | N |
| Hydrochloric 25%, 70 F | F |
| Hydrochloric 25%, 176 F | N |
| Hydrofluoric 48%, 70 F | E |
| Hydrofluoric 48%, 176 F | G |
| Lactic 5%, 70 F | G |
| Malic, all temps. | G |
| Nitric all concs., 70 F | N |
| Nitric 65%, boiling | N |
| Oleic all concs., all temps. | E |
| Oxalic 5%, boiling | G |
| Phosphoric 10%, 70 F | F |
| Phosphoric 85%, 70 F | P |
| Phosphoric 85%, boiling | N |
| Stearic concentrated to 200 F | G |
| Sulfuric 2%, 70 F | G |
| Sulfuric 2%, 176 F | F |
| Sulfuric 2%, boiling | G |
| Sulfuric 5%, 70 F | G |
| Sulfuric 5%, 176 F | F |
| Sulfuric 5%, boiling | G |
| Sulfuric 10%, 70 F | G |
| Sulfuric 10%, 176 F | P |
| Sulfuric 10%, boiling | F |
| Sulfuric 78% (60° Be), 176 F | N |
| Sulfuric 93% (66° Be), 70 F | F |
| Sulfuric 93%, 300 F | N |
| Oleum, 70 F | N |
| Mixed Acids 57% $H_2SO_4$, 28% $HNO_3$, 176 F | N |
| **ALKALIES** | |
| Ammonium Hydroxide, all concs. | P |
| Calcium Hydroxide Sat., 70 F | E |
| Calcium Hydroxide Sat., boiling | E |
| Sodium or Potassium, Hydroxide, all concs., 70 F | E |
| Sodium or Potassium Hydroxide < 20%, boiling | E |
| Sodium or Potassium Hydroxide 30% boiling | E |
| Sodium or Potassium Hydroxide, Molten, 600 F | G |
| **NEUTRAL AND ALKALINE SALTS** | |
| Barium Sulfide, 70 F | G |
| Calcium Chloride 5%, 70 F | E |
| Calcium Sulfate Sat., 70 F | E |
| Magnesium Chloride 5%, 70 F | E |
| Magnesium Sulfate 5%, 70 F | E |
| Sodium Carbonate, all concs., 70 F | E |
| Sodium Chloride 5%, 70 F | E |
| Sodium Sulfate 5%, 70 F | E |
| Sodium Sulfide 5%, 70 F | G |
| Sodium Sulfite 5%, 70 F | E |
| **ACID SALTS** | |
| Alum 10%, boiling | G |
| Aluminum Sulfate 10%, 70 F | G |
| Ammonium Chloride 5%, 70 F | G |
| Ammonium Sulfate 10%, 70 F | G |
| Ammonium Sulfate 10% boiling | G |
| Ammonium Nitrate, all concs., 70 F | E |
| Stannous Chloride 5%, 70 F | N |
| Zinc Chloride 5% boiling | G |
| **OXIDIZING ALKALINE SALTS** | |
| Calcium Hypochlorite 2%, 70 F | N |
| Sodium Hypochlorite 5%, 70 F | N |
| Sodium Peroxide | G |
| **OXIDIZING ACID SALTS** | |
| Ammonium Persulfate 5%, 70 | N |
| Cupric Chloride 1%, 70 F | N |
| Cupric Sulfate 10%, 70 F | N |
| Ferric Chloride 10%, 70 F | N |
| Ferric Sulfate, boiling | N |
| Mercuric Chloride 2%, 70 F | N |
| Stannic Chloride 5%, 70 F | G |
| **WET AND DRY GASES** | |
| Chlorine Gas Dry, 70 F | E |
| Chlorine Gas Wet, 70 F | N |
| Sulfur Dioxide Dry, 575 F | G |
| Sulfur Dioxide Wet, 70 F | F |
| Sulfur Dioxide Solution, 70 F | F |
| Sulfur Dioxide Spray, 70 F | F |
| Hydrogen Sulfide Dry | G |
| Hydrogen Sulfide Wet | G |
| **ORGANIC MATERIALS** | |
| Acetone, 70 F | E |
| Acid Sludge (50% $H_2SO_4$), 200 F | N |
| Alcohol—Methyl and Ethyl | E |
| Aniline Hydrochloride, 70 F | G |
| Benzol, 176 F | G |
| Carbon Tetrachloride | E |
| Chloroform | E |
| Ethyl Acetate, 70 F | E |
| Formaldehyde, 70 F | G |
| Phenol 5%, boiling | G |
| Refinery Crudes | G |
| Trichlorethylene, boiling | E |
| **PAPER MILL APPLICATIONS** | |
| Kraft Liquor | G |
| Black Liquor | G |
| Green Liquor | G |
| White Liquor | G |
| Sulfite Liquor, 176 F | N |
| Chlorine Bleach | P |
| Paper Makers Alum | G |
| **PHOTOGRAPHIC INDUSTRY** | |
| Humid Atmospheres | E |
| Cellulose Acetate | G |
| Acetic Anhydride | G |
| Acetic Acid + .1% $H_2SO_4$ | F |
| Developers | G |
| Solutions Containing $SO_2$ | G |
| Silver Nitrate, 70 F | N |
| **FERTILIZER MANUFACTURING** | |
| $H_3PO_4 + H_2SO_4 + HF$ | P |
| **PICKLING OPERATIONS** | |
| $H_2SO_4$ + Dichromate 176 F | N |
| $H_2SO_4$ + HCl, 176 F | F |
| **CORROSIVE WATERS** | |
| Acid Mine Water | F |
| Abrasive Acid Mine Water | N |
| Sea Water | G |
| Brackish Water | F |
| **FOOD & ASSOCIATED PRODUCTS** | |
| Brines | E |
| Edible Oils | E |
| Fats | E |
| Fatty Acid Distillation | G |
| Fruit Juices | G |
| Ketchup | F |
| Milk Pasteurizing | G |
| Vinegar and Salt, 70 F | F |

***RATINGS:***

*E—Excellent resistance. 0.001 max. in. per year of penetration. Corrosion so slight as to be harmless.*
*G—Good resistance. 0.001-0.012 in. per year of penetration. Satisfactory service expected; at most a slight etch.*
*F—Fair resistance. 0.012-0.120 in. of penetration per year. Satisfactory service under specific conditions. Light to moderate attack.*
*P—Poor resistance. 0.120-0.520 in. of penetration per year. Satisfactory for temporary service only.*
*N—No resistance. 0.520 min. in. of penetration per year. Rate of attack too great for any use.*
*†—Subject to pitting type corrosion.*

**No. 10**

(LAST OF A SERIES OF TEN)

# VACUUM TECHNOLOGY

## FROM ALL FIELDS

## FEATURES FRENCH LICK SYMPOSIUM

Players and kibitzers studying the entries before golf tournament starts.

VACUUM engineering in all of its diverse phases received a thorough-going technical appraisal last month at the French Lick meeting of the American Institute of Chemical Engineers. All the major engineering sciences using vacuum techniques were represented on the program, and engineers from all fields attended the meeting and helped in the discussion.

The program was interestingly thought out by Wheaton W. Kraft, vice-president of the Lummus Co., who managed, in the course of the three days, to plan papers on the theoretical aspects of vacuum engineering, from the mechanical engineers viewpoint, the use of vacuum in the process field, as well as much diversified information on the latest developments in the production of sub-atmospheres.

As usual the presentations were judged for performance by a subcommittee of a national program group, and top honors went to George J. Maslach, supervising engineer of the Institute of Engineering Research, University of California, for the delivery of his paper, "Vacuum Facilities for the Study of Supersonic Flow."

The three-day meeting purposely saw little of a formal social program. As typical with A.I.Ch.E. "resort meetings," this was part of a preconceived plan to allow the visiting engineers ample time to discuss technical problems with one another, and with authors. However, the engineers did have a golf tournament Tuesday afternoon as a

C. Kerby Stoddard, National Lead Co., South Amboy, N. J., asking a question.

William Burt, president, A.I.Ch.E., and vice-president mfg., B. F. Goodrich Chemical Co.

Mr. and Mrs. P. H. Groggins. (He is on A.I.Ch.E. Council.)

Group flinging questions at Georg Wilhelm Oetjen.

Left to right: D. M. Little, Phillips Petroleum Co.; V. V. Fondrk, Elliott Co.; Frank R. Hommowun, Worthington Corp.; W. E. Barr, Phillips Petroleum Co., and Elmer G. Ross, Worthington Corp.

Wayne Keller, of Mallinckrodt Chem. Wks., asking a question.

break in the long technical sessions. The winners were awarded prizes Tuesday night by the President of the Institute. William I. Burt, vice-president, B. F. Goodrich Chemical Co. First prize for low gross went to James S. Patterson of Pan American Refining Co., with second prize going to N. S. Eastman of Carbide & Carbon Chemicals Co., and third prize to Bruce Jones of Girdler Corp. The first three in the low net were G. Fevig of Upjohn Co., F. C. Baginski of Du Pont, and W. M. Leaders of Mallinckrodt Chemical Works.

## Technical Program

The technical program could conveniently be divided into three parts; first, a highly theoretical discourse on high vacuum; second, the aerodynamicists' use of high-vacuum systems; and third, practical application of vacuum engineering to the process industries.

The technical meeting opened with a paper by K. C. D. Hickman, consultant, Eastman Kodak Co., on the nature of liquid surfaces under vacuum. By means of slides, and motion pictures Dr. Hickman showed that the surface of many liquids evaporating in high vacuum ultimately separated into two parts, a torpid surface, or a skin-like covering from which relatively few molecules evaporated, and an active surface from which the vapor streamed at the fullest possible rate. Hickman aptly termed systems which showed this tendency as, schizoid.

G. J. Maslach, Institute of Eng. Res., Univ. of California, Berkeley.

F. M. Steele, The Hilliard Corp.

D. H. Jackson, of Croll-Reynolds Co.

Council in intensive study.

By means of the motion picture it was shown that each surface was not necessarily permanently in that form, and that transformation from torpid into an active surface occurred frequently during vacuum distillation.

Another paper in the theoretical field came from H. Bliss, A. M. Eshaya, and N. W. Frisch of Yale University who discussed rectification at reduced pressures. Much has been done in the field

(*Continued on page 42*)

# CARBIDE'S COAL HYDROGENATION PROCESS

ECONOMIC background for the new coal hydrogenation process of Carbide and Carbon Chemicals Co. was given last month in the news section (page 20) of Chemical Engineering Progress. This month's installment will give processing details.

The much-simplified flow chart on this page shows the beginnings of the two main raw material streams, hydrogen and coal. Both raw materials are amply available in the plant area. Bituminous coal abounds in West Virginia, and is trucked into the plant. Hydrogen is plentiful, since it is a by-product from dehydrogenation processes. Dr. George T. Felbeck, vice-president of the company, stated, in his discussion to the press, that there was enough hydrogen available in the Institute area to process 1000 tons of coal a day.

## Process Details

From the storage area coal is carried by belt conveyors to primary crushing equipment, and conveyed further after the initial pulverization to the coal preparation building. Here it may be stored temporarily prior to another crushing operation, this time to about the size of wheat grains. Proper size coal comes off the top of a classifier and goes to a dryer, where it is treated in a continuous stream of hot furnace gas, after which the dried coal is pasted with a recycle oil which, from the flow sheet, can be seen coming from the top of a recycle oil still. The paste is transferred by coal paste pumps at high pressure, to a preheater where it meets a stream of high-pressure hydrogen. Preheated hydrogen and coal pass into the converter continuously. It is in this vessel (theoretically) that the coal paste reacts with the hydrogen to form a predominantly liquid product.

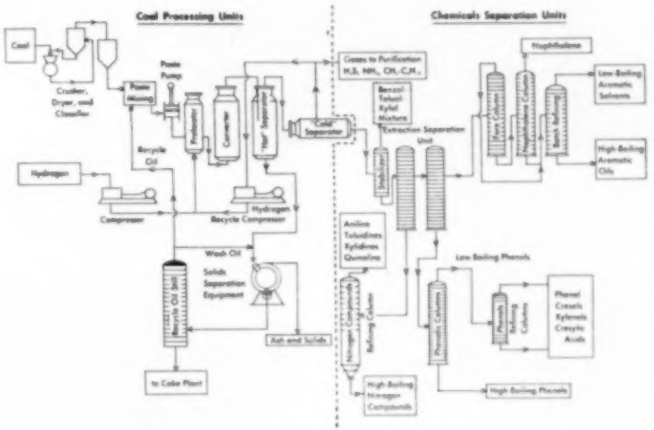
Operating temperatures and pressures are not yet fixed according to Carbide engineers, and they have still to find the proper pressures, temperatures, and throughputs for maximum efficiency. At the present time pressures are in the neighborhood of 4000 lbs./sq.in. and temperatures are as low as 300° C. Owing to the experimental nature of the plant, however, provision has been made to allow process operations up to 550° C. and pressures up to 6000 lbs./sq.in. Presumably also, according to Dr. Felbeck, the actual liquefaction of the coal takes place to the greatest extent during the mixing of the dried coal with the recycle oil, possibly owing to an exchange of hydrogen from the pasting oil.

From the high-pressure convertor the liquid is taken to a hot separator where a liquid and a vapor stream are split. The liquid stream containing high boiling oils, ash, and unreacted coal is reduced in pressure, and piped to a heavy products separation building. Here unreacted coal solids and ash are removed from the oil by solid-separation equipment which may involve centrifuging, filters, or flash vaporization. The solids removed from the heavy liquid product contain the ash of the coal, plus some carbonaceous material which represents portions of the coal which cannot be made to react with hydrogen under reasonable process conditions.

The liquid from the heavy oil stream is distilled in the recycle oil still into three fractions. The most volatile fraction is used as purge for the solid residue. The next higher boiling fraction (the major portion of the liquid product from the high-pressure convertor), is returned as recycle oil to the pasting operation. The third portion is the residue from the heavy liquid oil, which has potentialities as coke, or to quote the official source, ". . . is potentially useful for other products used by other divisions to supplement supplies of high carbon content raw materials."

(a) a hydrocarbon portion, (b) a phenolic portion and (c) an aryl amine and nitrogen heterocyclic portion.

The hydrocarbons are separated by distillation into various products, one of which, naphthalene, is purified further by crystallization. The phenolic portion is separated by continuous distillation into two fractions, one containing resin materials and the other containing higher-boiling phenolic materials. The crude hydrocarbon fractions and the crude phenolic fractions are further refined by various processes to obtain specification grades of aromatic solvents and resin raw materials. The nitrogen compounds portion is separated by batch distillation to yield aniline, toluidine, xylidines, quinolines, and other products.




Chemicals production by coal hydrogenation. Simplified flowsheet.

Returning to the hot separator unit the second product or vapor stream is condensed and passed to a cold separator. It is here that the unreacted gases are released. They are piped to a compressor building and the hydrogen is recycled to the preheater, the point where the pasted coal meets the high-pressure hydrogen for the first time. The other gases, such as hydrogen sulfide, ammonia, methane, butane, etc., are returned to Carbide's nearby plants for separation, use as fuel, or as chemical process raw materials.

The liquid stream containing the aromatic chemicals in which Carbide is interested, comes out of the cold separator, to a chemicals separation unit. This stream contains all the important recoverable products. These have been listed by the company as: toluol, xylol, naphthalene, methyl naphthalenes and higher hydrocarbons; cresols, xylenols, minor amounts of phenol and higher monohydric phenols, aniline, toluidines, xylidines, quinoline, methylated quinolines, indole, and other higher boiling nitrogen-containing chemicals. This stream is first broken into three principal fractions by chemical reaction and solvent extraction,

## Process Features

Carbide figures to break even on a 300-ton production, but the design of the plant is such that there is a possibility of upping capacity to 500-600 tons a day. This represents a first-class scale-up operation, since data were originally obtained on a laboratory scale unit of only ¼ ton a day. Other engineering details of great importance hinge around the Carbide reactor. Retention time is low. Since the German units for processing coal paste had a retention time of about 45 min., the construction of the Carbide reactor with its continuous quick-acting feature, may be the answer to the coking problem.

Another capital saving in the Carbide operation is the elimination of the high-pressure scrubber for unreacted hydrogen.

## NEW PETROLEUM CATALYST PLANT

**View of spray dryers of new petroleum catalyst plant constructed at Warrington, England, by Joseph Crosfield & Sons, Ltd., under technical direction of the Davison Chemical Corp., Baltimore, Md., whose processes are being used in the plant. The plant is expected to produce at the rate of more than 10,000 short tons of catalyst a year. The Davison process for producing the microspheroidal type of synthetic fluid silica-alumina petroleum cracking catalyst will be employed.**

## FIFTEEN FELLOWSHIPS IN GAS TECHNOLOGY

The Institute of Gas Technology, affiliated with Illinois Institute of Technology, Chicago, has 15 two-year fellowships for the training of men interested in utility gas. The fellowship program is designed to train men at master's and doctoral levels for positions of responsibility in the several phases of the manufactured and natural gas industries: production, distribution, utilization, administration, research and sales. The program was initiated in 1941 and has become the single source of graduate students specially trained in gas technology. The gas industry has created the educational and research facilities and assumed the financial burden for the fellows' tuition, fees, stipend, and summer plant training.

To a student working for a master's degree a fellowship currently is worth $4000 plus his earnings at summer employment in the industry. If he continues to a doctorate its value is increased by $5700 to a total slightly less than $10,000. Application forms may be obtained from the Dean of Students at Illinois Institute of Technology.

*(More News on page 40)*

## MINIMUM SALARIES FOR ENGINEERS SUGGESTED

A minimum salary for professional engineers, during the first quarter of 1951, of $7,200 to $8,400, the higher figure generally applying to the metropolitan area of New York City or where otherwise similarly applicable, has been recommended by the New York State Society of Professional Engineers, Inc. For Engineers-in-Training the recommended minimum salaries during the same period are $3,600 to $4,200, the higher figure generally applicable as in the first instance.

On the subject of fees the Society defines services included in minimum fees, services not included, fees on a lump-sum basis, consulting services, etc., though no penalties are proposed for non-conformance to the schedule.

These recommendations are contained in a 12-page brochure, titled Schedule of Minimum Fees and Salaries (1951), prepared by the above-named society. It is because inadequate compensation for professional services injures both the profession and the community that the New York State Society has adopted these schedules of minimum fees and salaries. The Society states it "expects the cooperation and good-will of all who perform or make use of engineering services."

## "C.E.P." WINS SAFETY AWARD

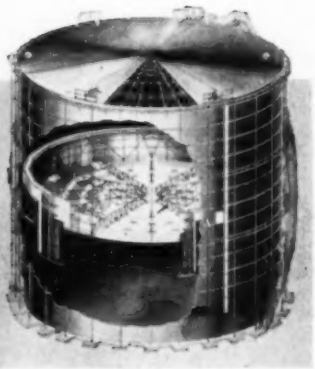
**Chemical Engineering Progress has been voted a National Safety Council's Public Interest Award for 1951. The Awards are made annually by the National Safety Council to public service media for exceptional service to safety. "C.E.P." won the honors for the series of articles it published on safety and which came from the safety symposium given last year at Kansas City. The symposium was planned and chairmaned by Mathew M. Braidech, research director, National Board of Fire Underwriters, New York, N. Y.**

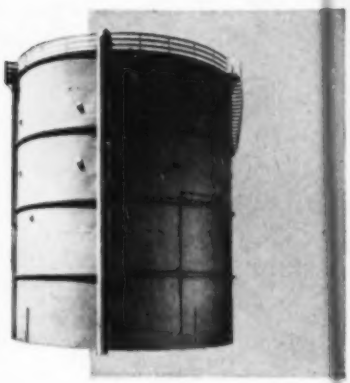
**Backing up the direct symposium on safety were articles on "Indoor vs. Outdoor Plant Installation," a symposium put together by Joseph R. Minevitch, of Badger Process Div. of Stone & Webster Eng. Corp., Boston, Mass., and which constantly stressed safety as an objective in chemical plant design.**

SIMPLICITY

EFFICIENCY

ECONOMY





# *Wiggins Gasholder* by GENERAL AMERICAN

. . . that's what you get when you specify WIGGINS GASHOLDERS for chemical process and industrial gases.

The absolutely dry, frictionless seal of the WIGGINS GASHOLDER is gas-tight and impermeable. Can't freeze or "hang up" —there's *no* water—*no* tar—*no* grease. *No* complicated mechanism. *No* gadgets. *No* operating costs. *No* maintenance costs.

Ask us about installations made from coast to coast for economy-minded process engineers—men who *know* that the Simplicity of the WIGGINS GASHOLDER in operation *always* results in Efficiency and Economy.

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# SPARKLER FILTERS

**Designed to use diatomaceous earth without the necessity of a fibrous material pre-coat to hold cake on the plate.**

With Sparkler horizontal plates there is no tensile strain on the cake. Even a very thin cake can be built up without danger of slipping or breaking as it rests in a horizontal position on the plate and requires no pressure to hold it in position. This saves considerable cost in pre-coating.

Sparkler horizontal filter plates can be removed for cleaning in one unit assembly and a clean set lowered into the filter tank immediately. This feature reduces down time to a matter of minutes.

***Representatives in all principal cities in the U.S.A.***

**SPARKLER MANUFACTURING CO.**
**Mundelein, Ill.**

Sparkler International Ltd.
Herengraht 568, Amsterdam, Holland

Sparkler Western Hemisphere Corp.
Mundelein, Ill., U. S. A.


## NEWS

(*Continued from page* 38)

### CONFERENCE ON CLAY TECHNOLOGY IN JULY

A National Conference on Clays and Clay Technology will be held at the University of California, Berkeley, July 21-25, 1952. The conference has been scheduled to bring together for an exchange of ideas and experiences men working in a variety of fields, having problems regarding clays and their uses. Morning sessions will begin at 9 a.m. and afternoon sessions at 2 p.m. On Tuesday evening at 8 p.m. there will be an open meeting of National Clay Minerals Committee with R. E. Grim, chairman of the committee. On Thursday evening at 8 p.m. visits to laboratories are scheduled.

### AMMONIA-UREA PLANT PROJECTED FOR MIDWEST

A more than $20 million ammonia-urea plant will be erected soon by W. R. Grace & Co., according to an announcement by the company's president, J. Peter Grace, Jr. It is understood that the site will be in the U. S. Middle West. The plant, with a daily capacity of 250 tons, will synthesize the ammonia from natural gas; part of this ammonia will be converted to urea. The products will be sold for agricultural and industrial use. The firm has had experience in the chemical field in South America. Its experience in the fertilizer field dates back to its founding nearly 100 years ago.

Plans already are underway for the integration of other petrochemical manufacturing operations into the new Grace plant.

### A.C.C.L. DIRECTORY NOW AVAILABLE

The 1952 revised edition of the Directory of American Council of Commercial Laboratories, Inc., is now available without charge upon request. This is a 73-page guide to the leading independent testing research and inspection laboratories of America. The issue contains a list of the members of the A.C.C.L., individual laboratory statements of organization, services, specialties, etc., a geographical index of member laboratories, branch laboratories, test stations and representatives, and a code of ethics which is an excerpt from A.C.C.L. Bylaws.

Copy may be obtained by addressing a request to the Executive Secretary, American Council of Commercial Laboratories, 4302 East-West Highway, Washington 14, D. C.

**CENTENNIAL SYMBOL**

**Above is the symbol to be used by all engineering societies cooperating in the Centennial of Engineering to be held this September in Chicago. The Centennial actually commemorates the one-hundredth anniversary of the American Society of Civil Engineers, and the A.I.Ch.E. is cooperating by having a meeting Sept. 11-13, at the Palmer House. Other engineering societies are also cooperating by holding meetings during the period of the Convocation.**

## MICROWAVE LINKS FREEPORT MINES

A microwave radio communication network, the first to link remote industrial operations with a metropolitan headquarters, will be used at the Louisiana sulfur mine of Freeport Sulphur Co. Approval for the system was given by the F.C.C., which said that the unprecedented installation was necessary because "no other communications system is feasible due to the terrain, marshes and high winds which make construction and maintenance of telephone lines impractical."

The license is the first private microwave grant to any company other than those engaged in electric power transmission or cross-country pipelines.

The microwave system, together with existing radio and radio-telephone facilities, will enable the company's southern headquarters at New Orleans to keep in touch with Grande Ecaille, its largest mine, 45 miles south of the city, and with new mining installations at Bay Ste. Elaine and Garden Island Bay, both in relatively inaccessible areas near the Gulf of Mexico.

Microwave transmission is ultra high frequency radio beamed to its destination through a series of relay towers the same as in television. The Freeport installation will make use of four manned transmitter stations—at New Orleans, Grande Ecaille, Bay Ste. Elaine and Garden Island Bay—plus four relay stations—at Port Sulphur, Myrtle Grove, Leeville and Venice. The license authorizes the use of six frequencies, and the system will have 24 voice channels.

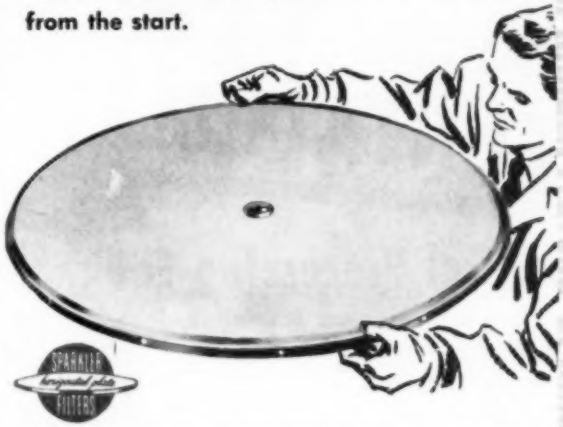
Cost of the system, estimated at between $100,000 and $150,000, is approximately half that of constructing telephone lines under normal conditions, and maintenance will be negligible because there will be no poles or wires to blow down in storms.

(*More News on page* 44)

SPARKLER FILTERS · SPARKLER FILTERS · SPARKLER FILTERS

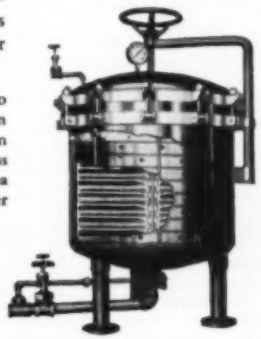
# SPARKLER FILTERS

**Designed for maximum pre-coat economy. Only a thin pre-coat cake is necessary to assure brilliant clarity of the filtrate right from the start.**




Sparkler horizontal plates permit the filter media to be floated onto the plate and deposited with gravity into a cake of uniform thickness and uniform density even though the first pre-coat is very thin. This saves considerable time and Filter Aid in pre-coating.

Since no pressure is needed to hold this thin pre-coat cake in position it is possible to begin filtering operation with less pressure which results in a cake of less density and greater flow rate.



***For personal service on your filtering problems, address correspondence to Mr. Eric Anderson***

**SPARKLER MANUFACTURING CO.**
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Sparkler International Ltd.
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Chemical Processing Equipment

"In making a high purity product such as ours, we can't take chances on picking up contamination from partly corroded equipment. Tests showed that tantalum is safe. Experience proved that tantalum is economical."

**TANTALUM,**
***The Acid-Proof Metal***

1. Acid-Proof means inert, not merely "resistant."
2. Full speed in heat transfer.
3. Freedom from thermal shock.

*Manifold type heat exchanger used in distilling c. p. HCl. One of these units has been in service since 1938.*

**USE TANTALUM WITH ECONOMY**
**for most acid solutions, corrosive gases or vapors; not with HF, alkalis or substances containing free $SO_3$.**

Acid-Proof TANTALUM

22405C

Fansteel Metallurgical Corporation NORTH CHICAGO, ILLINOIS, U.S.A.

## FRENCH LICK

(*Continued from page 36*)

of rectification, but the authors have found little to guide them in the theoretical consideration of the laws that govern separation of liquids under reduced pressures. Using a system of orthodichlorobenzene-orthodiethyl benzene, and a pressure of 16 to 100 mm. of Hg., and a ½-in. ring packing in the tower, the authors found that a fourfold variation of gas and liquid rates, had no appreciable effect on the height of transfer units.

As far as the use of vacuums in the study of supersonic flow, G. J. Maslach said that the simulation of high altitude flight in a wind tunnel would not be possible in aerodynamic studies if it were not for modern techniques in creating vacuums.

The low vacuums are necessary in order to create atmospheric conditions found at altitudes from 40 to 60 miles above the earth. According to Maslach, the availability of large-scale commercial vacuum pumps, capable of high capacity low density gas flow, has been the key to the solution of the problem. He stated:

> The problems associated with the design of vacuum structures, whether they be wind tunnels or stills, both require an understanding of structural design and sealing techniques, and instruments used to measure pressures in low density wind tunnels can be used to measure other pressures of low magnitude.

One of the problems that the engineers solved with a standard commercial ejector was the creation of supersonic flows. To produce such a jet 2-4 in. in diam., air flow rates had to be from 3 to 100 lbs. an hour at pressures of 30 to 300μ, Maslach revealed. Use of a five-stage steam ejector, it was found, provided an economical drive which would give a uniform superspeed jet of air for the wind tunnel tests. Maslach also described the special precautions necessary in building the wind tunnel to prevent leaks through welds and gaskets. Not only did the shell have to be completely tight, but special consideration had to be given to the metals out of which the tunnel was fabricated. Rolled or forged alloys were used since cast materials would be too porous at the vacuums used. Further, he pointed out that the formation of oxides or rust on the inside of the tunnel would create unfavorable test conditions since the oxide layer would take up moisture which would be emitted slowly over a long period of time. Low vapor-pressure paints and varnishes were used throughout the tunnel after the metal had been thoroughly cleaned. Maslach

(*Continued on page 44*)

**Y VALVES**

**ANGLE VALVES**

**SAFETY VALVES**

**PLUG COCKS**

**PIPE and FITTINGS**

## OF SOLID CHEMICAL PORCELAIN ARMORED WITH FIBERGLASS-REINFORCED PLASTIC

# Lapp TUFCLAD

# Lapp

## PROCESS EQUIPMENT

CHEMICAL PORCELAIN VALVES • PIPE • RASCHIG RINGS

PULSAFEEDER CHEMICAL PROPORTIONING PUMPS

**For** problems involving severe corrosion or freedom from metallic contamination, the chemical processing industry has found no more effective—or economical—material than Lapp Chemical Porcelain. Now this same solid porcelain material is available with TUFCLAD, a new *tough* armor which greatly adds to operating security—protection of personnel and equipment—certainty of avoiding product loss.

TUFCLAD is woven Fiberglass fabric, impregnated and bonded in multiple layers to the porcelain body with an Epoxide resin of high strength and chemical and heat resistance. Armor is tightly knit to porcelain and runs end-to-end, *under* flanges.

In operation, TUFCLAD provides a cushion to protect porcelain against accidental damage in handling or operation—and an insulator against thermal shock. But most important, the TUFCLAD shell is of itself homogeneous and *tough*—fully able to hold operating pressures against gross leakage even though porcelain is damaged by accident, explosion or fire.

Write for description and specifications of Lapp TUFCLAD-armored Porcelain valves, plug cocks, safety valves, flush valves, pipe, fittings and special shapes. Lapp Insulator Co., Inc., Process Equipment Division, 505 Maple Street, Le Roy, N. Y.

## FRENCH LICK

(*Continued from page 42*)

revealed that for gasketted joints, ordinary synthetic and natural rubber compounds were used.

F. M. Steele, research engineer, Hilliard Corp., reported to the delegates on the method of keeping oils, used in rotary high-vacuum pumps, clean and at the peak of performance.

Steele pointed out that there were several major means of contamination of the oil seal in rotary vacuum pumps. The oil itself may oxidize to form acids, or, in some cases, a varnish or lacquer may result. A second but relatively easily controlled contamination is due to careless handling on installation, while the third and most important contamination source comes from the material being processed. Examples of this latter are dust particles which become trapped in the oil film, condensation of solvents which have boiling points above the operating temperature of the pump, absorption of vapors by the oil film, and the slow eroding of metal parts by corrosive substances. As contaminants build up in the vacuum oil, pumping efficiency becomes less and less since the accumulation of volatile constituents increases the minimum pressure which the pump can develop in the process chamber.

Steele also pointed out that it was possible to replace the contaminated oil, but that such an operation either meant the shutdown of the process, or in the case of a continuous operation, the installation of duplicate standby equipment. Steele described new equipment for the removal of the contaminants which included removal of the dirty oil from the vacuum system, preheating it in a heat exchanger with clean oil going to the vacuum system, filtering the oil through fullers earth, after which the oil is passed through a vaporizer which removes the water, gases, solvents, etc. The oil then returns to the system via the heat exchanger.

Details of falling film evaporators were given by E. J. Kelly, of the Carrier Corp. The falling film evaporator, Kelly explained, concentrates liquids by dropping a thin sheet or film of material down the inside of large diameter tubes which are heated from the outside. The water vapor is removed from the liquid in a separate condenser. According to Kelly, the nature of the process is such that lower temperatures can be used in evaporation, thereby reducing corrosion of metals. Kelly showed data on the corrosion of mild steel by a salt solution in which the rate of corrosion was reduced 75% by the reduction of evaporation temperatures from 60° C. to 20° C. Since the falling film equipment works

**Bags packed, registrants wend their way homeward.**

under a partial vacuum, temperatures are less for a given liquid than they would be if the evaporation were carried on at atmospheric or increased pressures.

Another factor in preventing corrosion of metals is the reduction of the velocity of the treated liquors in passing through the evaporation tubes. Forced circulation through evaporators, he said, is one way of reducing the capital cost of equipment, and transfer surfaces, and of increasing heat transfer. However, increased velocities also speed up corrosion rates, and Kelly predicted a 75% reduction in corrosion rates by decreasing the velocity of circulating liquor from 9 ft. a sec. to 3 ft. a sec. Another advantage cited by the author was the better structural properties of certain materials at lower temperatures. "Chemical lead," he said, "will have an 80% increase in allowable fibre stress when decreasing temperatures from 130° C. to 40° C.

A method of plotting the characteristics of each type of high-vacuum pump on a single sheet of graph paper was shown at the meeting by R. B. Lawrence, director of the applied physics department, National Research Corp. The new technique of visualizing the data on a single chart was termed "synoptic," by the author.

The system consists of determining four important pressures, and charting them against the gas flow through the pump.

The pressures as listed by the speaker were: the inlet pressure to the vapor pump; the fore-pressure produced by the auxiliary vacuum system and against which the high-vacuum vapor pump exhausts; the tolerable forepressure defined as the pressure that causes a ten per cent increase in the inlet pressure; and the "breaking forepressure," a pressure which causes a tenfold increase in the inlet pressure to the pump.

By plotting these four values on graph paper against the quantity of gas handled by the pump, curves are obtained which visualize, in synoptic fashion, the pump's performance. This chart, Dr. Lawrence said, allows definite specifications and comparison of equipment of various types.

## KELLOGG TO BUILD OXO ALCOHOL PLANT

Standard Oil Company (Indiana) has named The M. W. Kellogg Co. to construct a large iso-octyl alcohol plant at its Wood River (Ill.) refinery, it has been announced by Kellogg. Standard had previously announced plans to construct the plant.

The plant is divided into three basic parts: a fractionation system for obtaining heptane from an olefin feed stock; a refinery gas reformer for preparation of hydrogen and carbon monoxide; and the actual iso-octyl alcohol section where the heptane, hydrogen and carbon monoxide are synthesized into the end product.

A primary use of iso-octyl alcohol, to be produced by the new plant at an annual rate of about 10 million pounds, is in the manufacture of plasticizers.

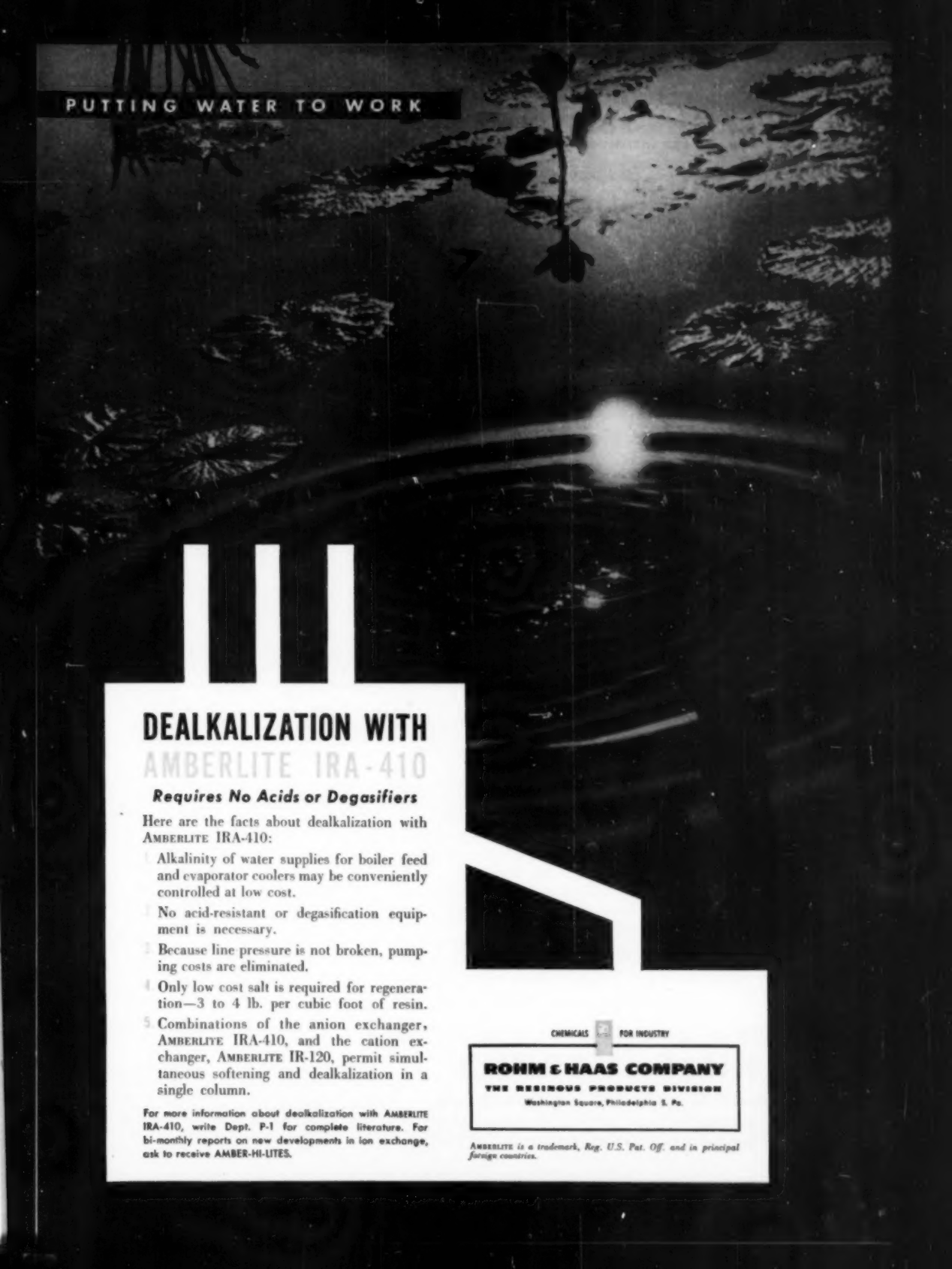
## DU PONT TO MARK 150TH ANNIVERSARY

The 150th anniversary of the establishment of the Du Pont Co. will be marked July 18 at the site of the first Du Pont powder mills on the banks of Brandywine Creek a few miles from Wilmington, Del. The ceremonies will include a simple historical dramatic prologue, addresses by company officials, and the dedication of a marker on the site of the first plant. About 6,000 spectators—representatives from the 71 plants and Wilmington offices, retired employees, members of the Du Pont family, and guests are expected.

Walter S. Carpenter, Jr., former president and now chairman of the board, will dedicate a marker formed by one of two huge millstones ordered from France by the founder of the company in 1801. The stone weighs about seven tons, and its function, by the application of water power, was to grind the ingredients of black powder—sulfur, charcoal and saltpeter. Bronze plaques designed and executed by Domenico Mortellito, Wilmington sculptor, will be mounted upon the stone, which in turn will rest upon a granite base.

The story of Du Pont's growth will be told in a book, "Du Pont—The Autobiography of an American Enterprise," to be published about the first of July.

(*More News on page 52*)




# DEALKALIZATION WITH AMBERLITE IRA-410

***Requires No Acids or Degasifiers***

Here are the facts about dealkalization with AMBERLITE IRA-410:

1. Alkalinity of water supplies for boiler feed and evaporator coolers may be conveniently controlled at low cost.
2. No acid-resistant or degasification equipment is necessary.
3. Because line pressure is not broken, pumping costs are eliminated.
4. Only low cost salt is required for regeneration—3 to 4 lb. per cubic foot of resin.
5. Combinations of the anion exchanger, AMBERLITE IRA-410, and the cation exchanger, AMBERLITE IR-120, permit simultaneous softening and dealkalization in a single column.

**For more information about dealkalization with AMBERLITE IRA-410, write Dept. P-1 for complete literature. For bi-monthly reports on new developments in ion exchange, ask to receive AMBER-HI-LITES.**

CHEMICALS FOR INDUSTRY

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**THE RESINOUS PRODUCTS DIVISION**

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*offers over 20,000 different "custom-made" assemblies on a standardized basis*

**32 standard operators**

Methods of operation for Hills-McCanna Valves include handwheel and lever operators, sliding stem, air and hydraulic cylinders, diaphragm motors and electric motors in a variety of types.

**14 standard diaphragms**

Diaphragms, the heart of Hills-McCanna Valves, are offered in the widest choice to best suit your individual needs; 5 types of rubber, 3 of Neoprene plus Hycar, butyl, Tygon, Compar, special polyethylene and Kel-F.

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By offering such a broad selection of operators, diaphragms and bodies, Hills-McCanna is able to furnish so many standard combinations that the user is in the position of being able, in most cases, to find the exact valve that best fits his individual requirements. There is virtually never any need to adapt to your service a valve developed for someone else's needs.

The 36 page catalog No. V-52 gives full details on all Hills-McCanna Valves. Write for your copy. HILLS-McCANNA CO., 2438 W. Nelson St., Chicago 18, Illinois.

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***Also Manufacturers of Proportioning Pumps Force Feed Lubricators • Magnesium Alloy Castings***


# MARGINAL NOTES

## *The Engineer and Verbalization*

**Communication of Technical Information. Robert M. Dederich. Chemonomics, Inc., New York. (1952) 116 pp. $5.00.**

*Reviewed by D. H. Killeffer, Consultant, Tuckahoe, N. Y.*

THIS little book attacks a point of serious weakness in the mutual understanding between technologists and laymen—the necessity to create a common ground for any communication between the two. Although an integral equation may contain the most exact and complete answer to a question asked by, let us say, a haberdasher, no engineer in his right mind would offer such a person a bald equation of that kind without first establishing a basis for his understanding of it. When the same kind of thing comes up in the regular course of an engineer's work and he is asked to report to his own management on some matter connected with his job, he too often assumes that the boss is just as familiar as he with the details and techniques of engineering. There communication fails and instead of an approving pat on the back the engineer is likely to get from higher authority only a cold and fishy "so what?"

Dederich attempts to analyze this aspect of the tremendously vital matter of communication for technologists and to suggest basic methods for improving what most of us admit is a bad situation. The problem of contact and understanding between highly expert and entirely lay persons is here discussed on a quite general level and on that account the argument suffers for the lack of illustrative examples. Perhaps, too, the comparatively condensed treatment of so large a subject in 116 small lithoprint pages crowded out examples that might well have been included.

## *Books Received*

**Modern Chemical Processes. Vol. II. Reinhold Publishing Corp., New York, N. Y. (1952) 299 pp. $5.00.**

**Principles of Geochemistry. Brian Mason. John Wiley & Sons, Inc., New York. (1952) 276 pp. $5.00.**

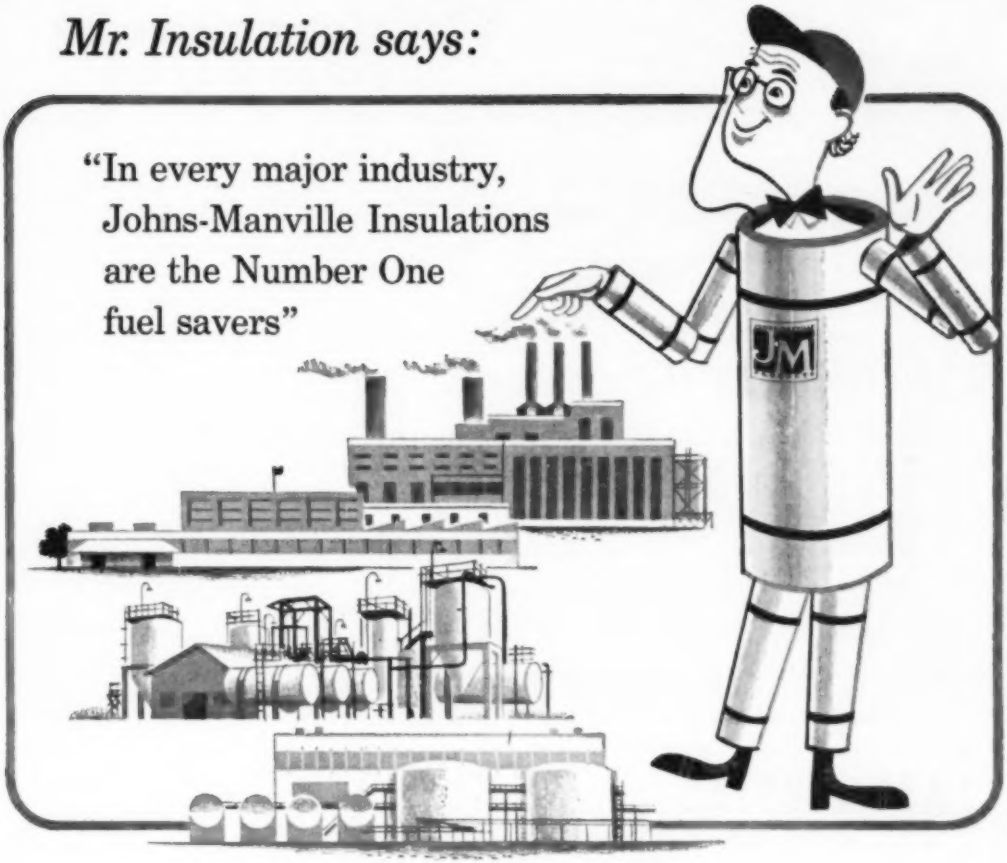
**Materials Technology for Electron Tubes. Walter H. Kohl. Reinhold Publishing Corp., New York. (1951) 493 pp. $10.00.**

**Color in Business, Science, and Industry. Deane B. Judd. John Wiley & Sons, Inc. (1952) 401 pp. $6.50.**

**Manufacturing Processes. Third Edition. Myron L. Begeman. John Wiley & Sons, Inc., New York. (1951) 608 pp. $6.00.**

**General Chemistry. Sixth Edition. H. G. Deming. John Wiley & Sons, Inc., New York. (1952) xii + 656 pp. $5.75.**

## *Mr. Insulation says:*




IN PLANTS with stacks or stills, tanks or towers, the story is the same—"Insulation by Johns-Manville" means the maximum return on your investment. There are two good reasons for this:

1. YOU GET THE RIGHT *MATERIALS*—From asbestos and other selected raw materials, Johns-Manville manufactures a wide range of industrial insulations for service from 400F below zero to 3000F above. If you need engineering advice in determining exactly the right one for your job, you can get experienced and authoritative assistance from your local Johns-Manville insulation engineer.

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Why not get the complete picture? Call on insulation headquarters when your next job is in the planning stage. Just write Johns-Manville, Box 60, New York 16, N. Y.

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***Low Cost Protection***
***for***

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In the past 31 years Conseco has provided millions of Flowrites for tube inlets of heat exchangers operating in all industries and all applications. Tube inlets become eroded and worn long before the remainder of the tube, and by reinforcing the inlets it is possible to increase the effective tube life many times.

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Steam, Hot Water, or Hot Oil circulated through Jacketed Piping and Fittings will do just that. Keep these otherwise lazy liquids on the move.

Parks-Cramer Jacketed Pipes come in sizes 1¼", 1½", 2", 3", and 4"—inside dimensions. Jacketed fittings include: two- and three-way Plug Cocks, Tees, 90° and 45° Elbows, Crosses, Expansion Joints and Relief Valves. Send for Bulletin 152C.

**Parks-Cramer Company**
ENGINEERS and CONTRACTORS
**Fitchburg, Massachusetts**


# CANDIDATES FOR MEMBERSHIP IN A. I. Ch. E.

The following is a list of candidates for the designated grades of membership in A.I.Ch.E. recommended for election by the Committee on Admissions.

These names are listed in accordance with Article III, Section 7, of the Constitution of A.I.Ch.E.

Objections to the election of any of these candidates from Active Members will receive careful consideration if received before July 15, 1952, at the Office of the Secretary, A.I.Ch.E., 120 East 41st, New York 17.

### APPLICANTS FOR ACTIVE MEMBERSHIP

Bell, F. Homer, *Atlanta, Ga.*
Bell, T. Robert, *Strafford, Pa.*
Black, David G., *Pataskala, Ohio*
Bowe, Leon E., *Webster Groves, Mo.*
Bright, B. K., *Kingsport, Tenn.*
Brown, Ernest S., *Tuckahoe, N. Y.*
Carey, James K., *Frederick, Md.*
Carr, William H., Jr., *F.P.O., San Francisco, Calif.*
Clark, Ezekail Louis, *Pittsburgh, Pa.*
Coleman, R. B., Jr., *Birmingham, Ala.*
Devore, Milton, *Glen Burnie, Md.*
Dotterweich, Frank H., *Kingsville, Tex.*
Egbert, Robert B., *Roslyn Heights, N. Y.*
Engisch, George W., *Linden, N. J.*
Goins, Robert R., *Bartlesville, Okla.*
Greiner, J. W., *Kalamazoo, Mich.*
Hernried, Erwin G., *San Francisco, Calif.*
Hoover, J. R., *Chagrin Falls, Ohio*
Jenks, Donald P., *Baltimore, Md.*
Jennings, C. M., Jr., *Charleston, W. Va.*
Kelly, Thomas B., *Charleston, W. Va.*
Kish, C. J., *N. Plainfield, N. J.*
Lieberman, B., *Charleston, W. Va.*
Lientz, Shannon D., Jr., *Chattanooga, Tenn.*
Linford, Henry B., *New York, N. Y.*
Long, John R., *Stow, Ohio*
Marshall, J. G., *Los Angeles, Calif.*
Martin, Joseph J., *Ann Arbor, Mich.*
Mayers, J. W., *Wyandotte, Mich.*
McCue, Robert B., *Charleston, W. Va.*
Morgan, W. Lewis, *Charleston, W. Va.*
Oney, James L., Jr., *S. Charleston, W. Va.*
Orr, Clyde P., *St. Louis, Mo.*
Owens, Albert P., *Louisville, Ky.*
Pierce, W. R., *E. Orange, N. J.*
Rose, B. L., *S. Charleston, W. Va.*
Rose, Chester E., *Metuchen, N. J.*
Rucker, John T., *Lewiston, N. Y.*
Scrantom, E. L., *Kingsport, Tenn.*
Shanley, W. B., *San Marino, Calif.*
Sommers, Alexander Arthur, *Brooklyn, N. Y.*
Temple, G. Franklin, *Paoli, Pa.*
Wolf, Van J., *Jackson, Mich.*

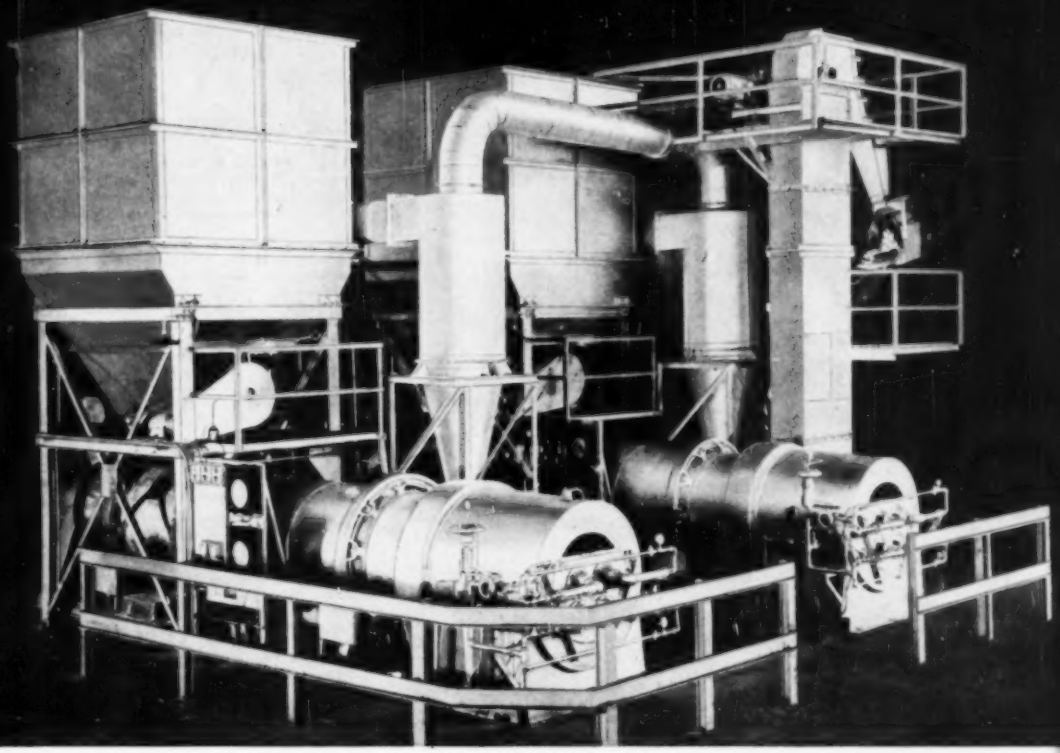
### APPLICANTS FOR ASSOCIATE MEMBERSHIP

Andrews, Robert V., *Richland, Wash.*
Atalla, Jorge Wolney, *Sao Paulo, Brazil, S. A.*
Morgan, Bernard R., *Fall River, Mass.*
Peters, Fred P., *New York, N. Y.*
Sundralingam, A., *Columbo, Ceylon*
Welsch, Harry W., *Belle, W. Va.*

### APPLICANTS FOR JUNIOR MEMBERSHIP

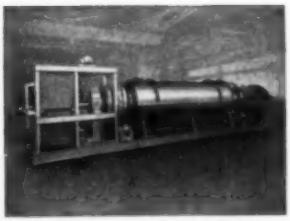
Baraga, John H., *Homestead, Pa.*
Bell, J. D., *Ballston Lake, N. Y.*
Bielecki, Edwin J., *Newtonville, Mass.*
Birch, F. Francis, *Wilmington, Del.*
Bockhoff, Frank J., *Cleveland, Ohio*
Bohn, Raymond T., Jr., *Harrisonburg, Va.*
Bowen, John C., *Bristol, Pa.*
Bree, Raymond F., *Bronx, N. Y.*
Brobst, Robert A., *Hiawatha, Kan.*
Buckel, James W., *St. Louis, Mich.*
Clarke, James A., *Lake Jackson, Tex.*

(Continued on page 50)



# compact *Twin Dryer* installation

## handles wide load fluctuations



Calciner and Cooler for Continuous Processing at Temperatures from 900°F. to 2100°F.

Special Stainless Steel Batch Dryer for Drying Fine Catalyst without Dust Loss.

● Consisting of two complete, self contained units, the twin systems pictured above can be operated together — or singly — depending upon plant requirements. Burners on each dryer are controlled separately, and automatically, for maximum fuel economy and to assure the discharge of a product having constant and uniform dryness regardless of variations in the rate, or moisture content of the feed. Highly efficient and thoroughly dependable, this is another example of Bartlett-Snow's skillful and detailed approach to every drying problem — your assurance that the diameter and length of shell, pitch, rate of feed and all other specifications of the equipment furnished will be exactly suited to your particular requirements. Let us work with *you* on *your* next job.

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*"Builders of Equipment for People You Know"*

CHECK YOUR *Sulfur Recovery*

by automatic measurement of $SO_2$ in vent gas

with this *continuously recording*

PLANT STREAM ANALYZER

**$ up the flue = $$ gone cloud-hunting . . .**

An accurate, continuous check on the percentage of $SO_2$ present in the vent gas is a simple first step toward reducing this needless loss of sulfur. In this application, this continuously recording Plant Stream Analyzer has a sensitivity of 0.01% concentration using a full-scale deflection of 1% on the recording indicator. Strong absorption of infrared by $SO_2$ assures high sensitivity to this compound in the dry state.

This is only one of the countless applications of this sensitive, reliable, sturdy instrument in control situations where the concentration of an infrared absorber in the process stream is a critical index of operation. In many cases, the adaptation of the instrument for automatic control of the process itself is realizing huge additional savings.

▶ WRITE TODAY FOR BULLETIN 36.

**PROCESS CONTROLS**

*a division of Baird Associates, Inc.*

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## CANDIDATES

(*Continued from page 48*)

Coalos, Andrew J., *Woodcliff Lake, N. J.*
Cohen, Edward S., *Lackawanna, N. Y.*
Crosnoe, Ted F., *Borger, Tex.*
Cutter, Louis A., *New Brunswick, N. J.*
DeBaun, Lester E., Jr., *Penns Grove, N. J.*
Donahue, Merrill L., *Pearl River, N. Y.*
Donald, J. Harry, *West Concord, Mass.*
Engelbrecht, Donald A., *Charleston, W. Va.*
Fairchild, William R., *Houston, Tex.*
Fleischmann, Alfred J., *Lawrence, Mass.*
Flynn, Clarence R., *Avon Lake, Ohio*
Foster, Joe S., *Huntsville, Ala.*
Frohman, Joseph H., *Houston, Tex.*
Frost, Gerard C., *Baden, Va.*
Garcia, Octavio, *Medellin, Colombia, S. A.*
Gardner, James H., *Cambridge, Mass.*
Graham, Ronald A., *Wyandotte, Mich.*
Greene, John T., *Bound Brook, N. J.*
Gutoff, Reuben, *Schenectady, N. Y.*
Hagerty, P. Frank, *Phila., Pa.*
Harmon, George W., *Takoma Park, Md.*
Hayton, B. C., *Beaumont, Tex.*
Hellwege, William H., Jr., *Brentwood, Mo.*
Hensch, Edward J., *Akron, Ohio*
Hill, Willace D., *Texas City, Tex.*
Howard, Robert W., *Nitro, W. Va.*
Inzerillo, Frank, *Brooklyn, N. Y.*
Jahnes, Henry J., *Willimantic, Conn.*
Krisher, A. S., *Texas City, Tex.*
Larson, Maurice A., *Midland, Mich.*
Leachty, Ernest W., *Lawrence, Kan.*
Marine, Gordon L., *Midland, Mich.*
Martin, Calvin L., *Williamsville, N. Y.*
McAdam, John R., *Niagara Falls, Ont., Canada*
McGlothlin, Bruce B., Jr., *Buffalo, N. Y.*
McKain, George D., *Louisville, Ky.*
McNeal, Allen E., *East St. Louis, Ill.*
Mink, William H., *Chicago, Ill.*
Nitshke, Norman L., *Lakeland, Fla.*
Pabich, H. L., *Argo, Ill.*
Perry, James H., *Pearl River, N. Y.*
Peterson, Rudolph A., *Wilmington, Calif.*
Rice, Richard D., *Schenectady, N. Y.*
Richards, John C., *Scotch Plains, N. J.*
Rinaca, U. S., Jr., *Harrisonburg, Va.*
Robinson, J. Wm., *Montreal, Que., Canada*
Ryan, Daniel W., *Midland, Mich.*
Schubert, David W., *Hammond, Ind.*
Sexton, Joseph R., *Baton Rouge, La.*
Simon, Frederick D., *Harrisonburg, Va.*
Simpson, Donald W., *Auburn, Mich.*
Sobala, Henry, *Houston, Tex.*
Soderberg, Richard W., *Midland, Mich.*
Starzmann, Howard A., *Oil City, Pa.*
Stewart, Robert H., *New Orleans, La.*
Tarika, Elio E., *Park Forest, Ill.*
Thomson, William P., *Whiting, Ind.*
Thrower, Herbert T., Jr., *Wilmington, Del.*
Tice, John D., *Woodbury, N. J.*
Toole, Robert L., *Louisville, Ky.*
Voskerchian, Sophia S., *New York, N. Y.*
Wainrib, Jules, *Alliance, Ohio*
Waters, Harvey K., *Harvey, Ill.*
Weil, Clifford M., *St. Louis, Mo.*
Wride, W. James, Bartlesville, Okla.
Yates, Edward A., *S. Charleston, W. Va.*
York, Philip K., *Richland, Wash.*
Zonis, Irwin S., *Belmont, Mass.*

# Secretary's Report

S. L. TYLER

THE Executive Committee met at the French Lick Springs Hotel May 11, 1952. The first item of business was approval of Minutes followed by receipt of the Treasurer's report for the months of March and April and also approval of bills. All those whose names appeared in "C.E.P." as approved candidates for membership were elected to the grades of membership as indicated. There were also 58 students elected to Student membership. F. Homer Bell and Fred W. Karl were appointed to the Membership Committee, the latter to replace L. G. Kline who had found it impossible to serve.

The appointment of J. S. Walton as Institute representative at the annual meeting of The Engineering Institute of Canada was confirmed. L. C. Kemp was appointed representative of the Institute on the U. S. National Committee of the World Power Conference. W. D. Kohlins and K. A. Kobe were appointed representatives with R. A. Morgen and S. L. Tyler as alternates to represent the Institute in the Engineers Joint Council delegation attending the meeting of the Pan-American Union of Engineering Societies at New Orleans in August, 1952. T. B. Drew was appointed representative on the U. S. National Committee on Theoretical and Applied Mechanics. W. N. Jones was appointed representative of the Institute on the Engineers' Council for Professional Development for a three-year term beginning Oct. 1, 1952.

Two resignations from membership were accepted and one member, Byrd Hopkins was placed on the Suspense List because of his having entered the Armed Forces. The Secretary reported that 49 Junior members had passed their thirty-fifth birthday and had discontinued their membership in the Institute.

The Council of the Institute met at the French Lick Springs Hotel on the afternoon of May 11, 1952, with twelve members present. The Minutes of previous meetings were received and approved and actions of the Executive Committee were confirmed.

A report was presented for the Research Committee by G. T. Skaperdas in the absence of W. E. Lobo, the chairman. He reported that $60,000 had been promised for the research program and that matters were progressing satisfactorily. Tentative arrangements have been made for research on *Bubble Cap Performance* with several universities

(*Continued on page 52*)


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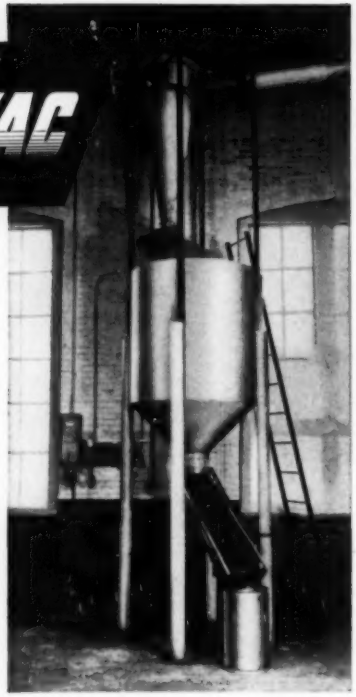

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PNEUMATIC CONVEYORS

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## SECRETARY'S REPORT

(*Continued from page* 51)

and it is expected that the actual work will be in progress during the summer.

A report was received from L. W. Bass, chairman, Special Committee on Unity in the Engineering Profession, and the suggestion was approved that the Institute encourage the Engineers Joint Council in the modifying of the Constitution to provide for enlargement and acceptance of the general recommendations of the Unity Committee.

The holding of a national meeting of the Institute in Washington, D. C., in February, or March of 1954, was approved.

The authorization for the formation of a student chapter at the University of New Hampshire was granted on recommendation of the Student Chapters Committee following petition from the student group there.

The Auditor's Report for 1951 was received and approved and ordered published.

The Council of the Institute had before it for some time a proposal for special membership coverage by Health and Accident Insurance on a Group Plan. After careful study the Council did not accept the proposal.

Secretary reported the receipt of $3,000 as a bequest by the late A. E. Marshall, income from which is to be used for a student prize.

### 1952 Year Book

Please add to the list of accredited curricula on page 9, the University of Detroit, Detroit, Mich., and the Missouri School of Mines and Metallurgy, Rolla, Mo., accredited as of the year 1951.

## CORROSION FILM OF INTERNATIONAL NICKEL

The International Nickel Co., 67 Wall Street, New York 5, N. Y., has recently released for the use of schools, colleges, technical societies, industrial plants, etc., a 16-mm. color and sound film entitled "Corrosion in Action."

## CHEMICAL PRODUCTION TELECAST BY N.B.C.

The National Broadcasting Co. selected J. T. Baker Chemical Co., Phillipsburg, N. J., to dramatize high purity chemical manufacturing on the weekly television news reel, "Industry On Parade," shown coast-to-coast. The film, about five minutes in length, traces the manufacture of potassium sulfate. Theme of the program is how high purity standards are maintained. The reel shows pictures of the company's "three-million dollar expansion program," and "dust removal system which guards the health . . . . of employees."

(*More News on page* 59)

## FUTURE MEETINGS AND SYMPOSIA OF A.I.Ch.E.

*Chairman of the A.I.Ch.E. Program Committee*

Walter E. Lobo, The M. W. Kellogg Co., 225 Broadway, New York 7, N. Y.

### MEETINGS

Chicago, Ill., Palmer House, Sept. 11-13, 1952.
*Technical Program Chairman:* D. A. Dahlstrom, Chem. Eng. Dept., Northwestern University, Evanston, Ill.

Annual — Cleveland, Ohio, Hotel Cleveland and Carter Hotel, Dec. 7-10, 1952.
*Technical Program Chairman:* R. L. Savage, Dept. of Chem. Eng., Case Inst. of Tech., Cleveland 6, Ohio.

Biloxi, Miss., Buena Vista Hotel, Mar. 8-11, 1953.
*Technical Program Chairman:* Norman A. Spector, Vitro Corp., 233 Broadway, New York 7, N. Y.

Toronto, Canada, Royal-York Hotel, April 26-29, 1953.
*Technical Program Chairman:* Brymer Williams, Dept. of Chem. and Met. Eng., University of Michigan, Ann Arbor, Mich.

San Francisco, Calif., Fairmont and Mark Hopkins Hotels, Sept. 13-16, 1953.
*Technical Program Chairman:* R. W. Moulton, Head, Dept. of Chem. Eng., University of Washington, Seattle, Wash.

Annual—St. Louis, Mo., Hotel Jefferson, Dec. 13-16, 1953.
*Technical Program Chairman:* R. M. Lawrence, Monsanto Chem. Co., St. Louis 4, Mo.

### SYMPOSIA

**Distribution of Chemicals**
*Chairman:* D. A. Dahlstrom, Northwestern University, Evanston, Ill.
*Meeting*—Chicago, Ill.

**Monobed Ion Exchange**
*Chairman:* F. J. Van Antwerpen, Editor, Chemical Engineering Progress, 120 East 41st St., New York 17, N. Y.
*Meeting*—Chicago, Ill.

**Applied Thermodynamics**
*Chairman:* W. C. Edmister, California Research Corp., Richmond, Calif.
*Meeting*—Cleveland, Ohio

**Human Relations**
*Chairman:* R. L. Demmerle, General Aniline & Film Corp., 230 Park Ave., New York, N. Y.
*Meeting*—Cleveland, Ohio.

**Modern Statistical Methods in Chemical Engineering**
*Chairman:* C. Daniel, Engineering Statistician, 116 Pinehurst Ave., New York 33, N. Y.
*Meeting*—Cleveland, Ohio.

**Filtration**
*Chairman:* F. M. Tiller, Dir., Div. of Eng., Lamar State College of Technology, Beaumont, Tex.
*Meeting*—Cleveland, Ohio

**High Pressure**
*Chairman:* E. W. Comings, Head, School of Chem. & Met. Eng., Purdue University, Lafayette, Ind.
*Meeting*—Cleveland, Ohio.

**Mining, Metallurgical Techniques for Chemical Engineers**
*Chairman:* N. Morash, Titanium Div., National Lead Co., South Amboy, N. J.
*Meeting*—Biloxi, Miss.

**Chemical Engineering in Hydrometallurgy**
*Chairman:* John D. Sullivan, Battelle Memorial Institute, Columbus, Ohio.
*Co-Chairman:* John Clegg, Battelle Memorial Institute, Columbus, Ohio.

**Fluid Mechanics**
*Chairman:* R. W. Moulton, Head, Dept. of Chem. Eng., University of Washington, Seattle, Wash.

**Absorption**
*Chairman:* R. L. Pigford, Div. of Chem. Eng., Univ. of Delaware, Newark, Del.

Authors wishing to present papers at a scheduled meeting of the A.I.Ch.E. should first query the Chairman of the A.I.Ch.E. Program Committee, Walter E. Lobo, with a carbon copy of the letter to the Technical Program Chairman of the meeting at which the author wishes to present the paper. Another carbon should go to the Editor, F. J. Van Antwerpen, 120 East 41st Street, New York 17, N. Y. If the paper is suitable for a symposium, a carbon of the letter should go to the Chairman of the Symposia, instead of the Chairman of the Technical Program, since symposia are not scheduled for any meeting until they are complete and approved by the national Program Committee. Before authors begin their manuscripts they should obtain from the meeting Chairman a copy of the Guide to Authors, and a copy of the Guide to Speakers. The first book covers the preparation of manuscripts, and the second covers the proper presentation of papers at A.I.Ch.E. meetings. Presentations of papers are judged at every meeting and an award is made to the speaker who delivers his paper in the best manner. Winners are announced in *Chemical Engineering Progress*, and a scroll is presented to the winning author at a meeting of his local section. Since five copies of the manuscript must be prepared, one should be sent to the Chairman of the symposium and one to the Technical Program Chairman of the meeting, or two to the Technical Program Chairman if no symposium is involved and the other three copies should be sent to the Editor's office. Manuscripts not received 70 days before a meeting cannot be considered.


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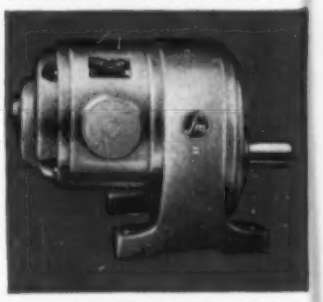

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*70 ILLUSTRATIONS* showing how Sterling Electric Power Drives reduce production costs. Write for Bulletin No. C-120.

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Chemical Engineering Progress

# DATA SERVICE

## EQUIPMENT

*1 • PORTABLE pH METER.* A battery-operated pH instrument which may be carried in the plant for use independent of outside power circuits has been developed by Beckman Instruments, Inc. Electrodes plug in side of instrument case, and are quickly interchangeable. Covers complete pH range. Built-in temperature compensation from 0 to 100° C, accuracy to .05 pH unit, weighs 8 lbs. Available with or without carrying case.

*2 • GYRATORY SEPARATOR.* Southwestern Engineering Co. has a unit for wet or dry separation with multi- or single-deck vibrating screens. Circular construction. The vibration is accomplished by a combination of two eccentric weights, on the upper and lower ends of motor shaft. Relationship between weights cause horizontal and vertical vibration. Can be mounted on top of bins, upper floors of buildings, 48 in. in diam., uses a 1-hp. motor, screen cloth can be changed quickly.

*3 • SLURRY VALVE.* For proportioning and controlling slurries, particularly pulp and paper stock, Fischer & Porter Co. has a new Ratogate valve for low-pressure automatic service. Operating range of 15 to 1. Has diamond-shaped outlet and is self-cleaning. Valve swept clean by agitating action of the flow passing through the body. Sight-glass window permits inspection. Equipped with pneumatic cylinder operators and positioners. In stainless steel and other materials.

*4 • SAFETY FAN.* For removing dangerous or obnoxious fumes, dirt, heat, smoke, etc., Standard Electric Mfg. Co., Inc., is making a general purpose fan with the motor outside the air stream. Explosion-proof enclosed, or open-type motors available. Belt drive. Meets Underwriters' specifications for spray booths. Cast aluminum fan blade 18 to 42 in. Motors from ¼ to 7½ hp.

*5 • DIAL INDICATOR.* For measuring temperature, pressure, load applications and other industrial variables, Taylor Instrument Companies have a newly designed 6-in. dial indicator for modern instrument panels. Micro-adjustment to permit zero settings, stainless steel movement to avoid linkage errors. For variables using a Bourdon spring as an energy-to-motion converter. For temperature applications four types of actuation—mercury, vapor, gas, or organic liquid. T range from −100 to +1200° F. Pressure range from 12 to 20,000 lbs./sq.in. Tension and compression load ranges from 0 to 75,000 lbs.

*6 • GRAPHITE COMBUSTION CHAMBER.* Graphite combustion chamber using graphite burner nozzles, new with National Carbon Co. Chambers are vertical graphite towers externally water-cooled and designed for burning wet hydrogen and chlorine gases at temperature reaching 5000° F. Natural gas, methane, steam can also be burned. Catalog available which describes equipment, installation, and operation, capacity, sizes and list of related equipment such as absorbers, pumps, valves, etc.

*7 • VIBRATORY TABLE.* A vibrating table for many uses by Cleveland Vibrator Co. For packaging small parts, granular materials, settling molds, filling containers with viscous liquids, etc. Uses air as actuating material. Intensity of vibration can be varied, permitting operation at 20 to 100 lbs./sq.in. line pressure. Vibrator controlled by knee valve. Table top 12 in. x 18 in. x 1½ in., floats on steel springs.

*8 • CYCLOTHENE PIPE.* A semiflexible piping resistant to solvents, mineral acids, salts, alkalies, etc., at temperatures up to 150° F. is being made by Munray Products, Inc. Data sheet from company gives physical properties, chemical resistance. Unaffected by temperatures as low as −50° F. Safe for transportation of pharmaceuticals or food. In sizes from ½ in. to 6 in. in stock lengths from 25 ft. for 4 in. and larger pipe, to coils of 400 ft. for smaller sizes. Flexibility allows it to follow surface contours. Connected with knurled and inset connectors clamped with stainless steel bands.

*9 • VACUUM PUMP.* For plant laboratory or research processes where high vacuum is required Central Scientific Co. has a new Hypervac pump, free air displacement of 41 l./min. and a pumping speed at 1$\mu$ at 0.35 l./sec.

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*10 • POURING SPOUT.* A pouring spout for carboys with a safety air vent to prevent spurts is new with the General Scientific Equipment Co. Made of acid-resisting rubber and plastic tubing, has a capacity of 5 gals./min.

*11 • COMPACT PUMPS.* By using standard mechanical shaft seals and shorter shaft lengths, Peerless Pump Division of Food Machinery & Chemical Corp. is offering a new line of horizontal split-case, general service pumps which conserve floor space. Available in discharge sizes from 1½ in. to 4 in. Capacities up to 750 gals./min. with a head range up to 230 ft. All types of drives. Electric, gasoline engines, steam turbine. Handles liquids up to 200° F.

*12 • POLYETHYLENE PIPE FITTINGS.* Being manufactured by American Agile Corp., a line of pipe fittings, tees, crosses, elbows, etc. up to 6 in. nominal pipe size from polyethylene. Fully welded construction.

*14 • STAINLESS STEEL CONDENSERS.* Standard steel vapor condensers by Doyle & Roth Mfg. Co. Two basic designs for condensing vapors on the tube and shell sides. Effective square foot of surface on the shell model varies from 62 to 317 sq. ft., nominal tube lengths from 48 to 72 in. and tubes vary in number from 26 to 40. On the vapor tube model, effective square feet of surface varies from 52 to 350 sq. ft., tube lengths from 48 to 72 in. and number of tubes from 26 to 40.

*15 • EXPLOSION-PROOF INTERCOM.* For the chemical plant with need for communication between hazardous areas, Executone, Inc., has a new explosion-proof intercom system. Especially designed for work areas containing inflammable and explosive gases, vapors, or dusts. Features a remote reply device which enables workers to answer without manipulating switches or going to the instrument. Bulletin describing setup, wiring systems, etc.

*16 • STAINLESS STEEL FOG NOZZLE.* From Bete Fog Nozzle, Inc., a new line of low-priced, stainless steel fog nozzles. Smaller in size and lighter in weight than previous models. Employ spiral principle of atomization. Available in six models, three flow rates 1-10 gal.

*17 • POROUS STAINLESS STEEL.* Micro Metallic Corp. new high tensile, smooth surface, porous stainless steel sheet. For use in the chemical processes which require release of collected solids. Applications would include rotary vacuum filters cleaned by blow back, vacuum plate filters, fluid catalyst filters, etc. Flow capacities range from 200 cu.ft./min. of air a sq. ft. of filter surface at 10 lb./sq.in. differential pressure, or from 20 gal./min. a sq. ft. of water at 10 lb./sq.in. differential. Pore diameters range from 20$\mu$ to 0. Various stainless alloys available including Monel and Stellite.

*18 • CHLORINE VAPORIZER.* For converting liquid chlorine to dry vapor Whitlock Mfg. Co. new vaporizer for use in pulp, textile, chemical, sewage, and industrial waste plants. Capacity of 2000 lb./hr. of chlorine with steam at 5 lb./sq.in. gage. All steel construction, easily disassembled and cleaned.

## CHEMICALS

*20 • ACRYLAMIDE.* From American Cyanamid Co. a new acrylic monomer Acrylamide. A crystalline product easily polymerized and copolymerized for application in adhesives, dispersing agents, thickening agents, synthetic leathers, and rubbers and for approving the affinity of acrylonitrile fibers for acid dyes. Has a reactive double bond which permits addition of alcohols, amines, etc. Bulletin available.

*23 • SODIUM DISPERSIONS.* A technical bulletin from National Distillers Chemical Co., a fourth in a series on sodium dispersions. Includes data on the formulation of the dispersions. Gives technical data of various media varying from toluene, xylene through kerosenes, light naphtha, paraffins, petroleum jelly, etc. Dispersions of about 50% sodium by weight. Gives particle size in microns, dispersant, stability, etc.

*24 • CHEMICAL PRICE LIST.* From The Matheson Co., Inc., a new price list of 189 commercial chemicals offered in 5-gal. units. The price list also gives the name of the manufacturer

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of the chemical, the specifications, and the net weight in pounds. For the chemical manufacturer who requires a small supply of commercial materials.

*25 • ETHYLENE CARBONATE.* Jefferson Chemical Co., Inc., has begun manufacture of ethylene carbonate in pilot plant quantities. Technical bulletin, samples available. Material is odorless, colorless, low-melting solid, nonhygroscopic and noncorrosive. For use as a solvent for acrylonitrile polymers, lignin, nitrocellulose, acetate, etc. Reacts with aliphatic amines to form carbonates. Thought by company to offer a new route to the preparation of agricultural chemicals, resins, fungicides, etc.

*26 • FLEXOL CC-55.* From Carbide and Carbon Chemicals Co. Flexol placticizer CC-55 in tank car quantities. It is di-2-ethylhexyl hexahydrophthalate and is a primary plasticizer for vinyl chloride resins. Used for calendered and extruded goods as well as organosols and plastisols. Technical bulletin gives physical properties, compatibility with various resins, description of its uses, color stability, characteristics of resins, etc.

*27 • WET-STRENGTH RESIN.* From Hercules Powder Co. a folder describing the advantages of using Kymene for obtaining better wet-strength paper. Folder gives the results of data from tests on bag paper compared to a government specification. Kymene is a strongly cationic resin in water solution. Uses no acid or aging ingredients and is unaffected by high sulfate ion concentrations.

*29 • LUBRICATING OIL ADDITIVE.* A lubricating oil additive combining antioxidant and bearing corrosion inhibition properties with highly effective alkaline detergency is announced by the American Cyanamid Co. Called Aerolube 92, it is specifically designed for Series 2 oils where high-sulfur fuels are used or in high-output supercharged equipment. Data sheet giving chemical and physical properties available.

*30 • GLUCURONIC ACID.* Corn Products Refining Co. is making glucuronic acid in the lactone form in commercial quantities. For uses as a solubilizing agent for insoluble drugs, as a diuretic in the treatment of allergies, in the pharmaceutical industries as a gut sterilizing agent, as a growth factor, etc.

*31 • CATION EXCHANGE RESIN.* A new low-cost medium capacity cation exchange resin, Amberlite IR-112, is new with Rohm & Haas Co. Stable up to 250° F. over an entire pH range it is characterized by high porosity. Has exchange characteristics in both hydrogen and sodium cycle operation. For use in split stream dealkalization for boiler feed, for bottling, and other processes requiring soft, dealkalized water. Its porosity prevents pore blocking by calcium sulfate. Data available in technical bulletins.

*32 • DRIERS.* Technical information on calcium naphthenate, calcium tallate and calcium octoate from the Nuodex Products Co., Inc. For use as auxiliary driers in paints and varnishes. Bulletin.

*33 • OXIDIZING VAT DYES.* Buffalo-Electro-Chemical Co., Inc. bulletin on oxidation of vat dyes on cotton and synthetics with hydrogen peroxide. Covers typical procedures for jig and packaging machines.

*34 • VALVE CLEANER.* A heavy-duty alkaline cleaner, Magnus 61RS from the Magnus Chemical Co., Inc., for cleaning valves and fittings. Deposits removed consist of oil, paint, lacquer, carbon, etc. Cleaner has no fumes. Valves and fittings cleaned by loading them into heated tanks.

*35 • ESTERS.* A new booklet on esters published by Carbide and Carbon Chemicals Co. Describes more than 30 esters, gives their uses, physical properties, and shipping data. Booklet of 36 pages also describes solubility of various resins in some of the esters as well as their performance in nitrocellulose lacquer. Includes a bibliography and a section covering specifications.

*36 • ION EXCHANGE.* Several bulletins on ion exchange materials made by National Aluminate Corp. Bulletin (57) covers Nalcite SAR, a strongly basic anion exchanger, (58) covers Nalcite HCR, a styrene-type cation exchanger, and (59) Super Nalcolite, a synthetic gel-type zeolite for cation exchange. The books give physical properties, expansion data, regeneration and capacities, plus the effects of various pH's, sodium salt, etc.

*37 • ISOBUTYL ACETATE ALCOHOL.* A comparison of the properties of isobutyl acetate and isobutyl alcohol made from isobutyraldehyde with normal butyl homologues with respect to lacquer formulation is the feature of a booklet now published by Tennessee Eastman Co. The comparisons are based on characteristics such as relative evaporation rates, dilution ratios, the effect upon viscosity in resin solutions. Covers application to government specification lacquers also.

*38 • $CO_2$ USES.* A wall chart showing the industrial uses of dry ice and $CO_2$ gas. From the Liquid Carbonic Corp. The chart is keyed with double references for the industry and the use.

*39 • SYNTHETIC RESINS.* A technical bulletin from Resistoflex Corp. on a number of corrosion-resistant and flexible synthetic materials including fluorocarbons. Covers Teflon and Kel-F, Compar and Silicone Rubber. For the first two the application of the materials to engineered parts is covered plus physical properties, linear expansion, dielectric constants, etc. Compar is a modified polyvinyl alcohol for use with oils, fuels and water insoluble organic chemicals. Silicone rubber has good thermal stability over a broad range.

*40 • DRESINATE.* Hercules Powder Co. with a technical bulletin describing properties and application of

Dresinate a series of sodium and potassium salts of rosins and resins. Covers use of materials for emulsifying soluble oils, extra-action alkaline cleaning compounds, emulsifier in textile oil emulsions, soaps, detergents. Gives complete technical data, compositions, grades, properties, formulations, etc.

*41* • *PETROLEUM PRODUCTS.* A brochure on specifications and technical data on petroleum products of the Pennsylvania Refining Co. Covers light oils, petrolatums, petroleum sulfonates, solvents, etc. Specifications & descriptions.

## BULLETINS

*42* • *PROCESS MACHINERY.* Hardinge Co., Inc., has a new 8-page bulletin describing complete line of process machinery for mining, chemical, stone, ceramic, mineral, paper, etc., industries. Includes oil-water separators, heavy-media separators, flocculating equipment, etc.

*45* • *INSTRUMENT CATALOG.* Some 20 different instruments and basic accessories of Beckman Instruments, Inc., are described in a new catalog. Covers pH meters, glass electrodes, spectrophotometric equipment including a model which permits continuous automatic analysis of flowing plant streams of gases or liquids. Other sections describe automatic titrators, micro-microammeters, flow colorimeter, and radioactivity instruments.

*46* • *CRUSHERS.* A line of custom-built crushers, grinders, shredders and choppers from the American Pulverizer Co. Various models are described including a high tonnage rolling-ring crusher which can be obtained from 40 to 200 hp. Other smaller models are available in different units. Hammermills, laboratory mills, knife choppers, etc., are all described.

*47* • *DEWATERING PRESS.* A large-capacity continuous dewatering press for removing bulk water from semi-solids. Bulletin from the P & L Welding & Machine Works, Inc. For dewatering paper, distillers grains, beet pulp, foods, etc. Can be used prior to a heat evaporation. Will dewater any material fibrous enough to pack. Solids leaving press have about 1 to 2% moisture.

*48* • *BULLETIN CHECKLIST.* A bulletin of bulletins published by the International Nickel Co., Inc. A 12-page listing of available literature on the various alloys made by the company. Data on corrosion, chemicals, petroleum, leather, and food processing industries, etc. Contains coupon to order literature.

*49* • *PHOTOCHEMICAL EQUIPMENT.* A general installation and operating manual for Hanovia photochemical equipment. Features ultraviolet radiation units for speeding up reactions such as hydrogenation, oxidation, reduction, decomposition, polymerization, bleaching, halogenation, etc. Described are immersion well assemblies, plus description and installation procedures for all the equipment used with it. Equipment available in wattage from 1200 to 4250.

*50* • *CASCADE COOLERS.* A bulletin describing a unit made by Corning Glass Works of Pyrex glass for heat exchange by the cascade principle. Bulletin describes the construction, the thermal conductivity of the Pyrex glass, corrosion resistance, etc. Units can be had with glass pipes varying from 1½ in. to 3 in. and coolers with as many as 40 tubes in a single unit can be built. Smooth surface of the glass resists formation of scale deposits which impede heat flow. Maximum allowable working pressures vary from 20 lbs./sq.in. for 3-in. pipe to 35 lbs. for the 1½-in. Construction details showing adapter connections, flared ends of the heat-exchanger pipe, standard flange assembly are all shown. Utilizes a trough principle to distribute and collect cooling liquid. Illustrated, gives parts list as well as nomographs and tables on how to determine capacities and requirements of a heat exchanger.

*51* • *HEAT EXCHANGERS.* Bell & Gossett Co. bulletin on Hydro-Flo shell and tube heat-transfer equipment. Shows standard design of equipment. 125 lb. pressure limits, shell diameters from 4 to 24 in., tube sizes from ½ to ¾ in. Shows construction, tube sheets, preheater, suction heaters, evaporators, refrigeration equipment, centrifugal pumps, plus a section of engineering data, tables giving fouling resistances, U factors for shell and tube units, film coefficients, conversion factors, physical properties of various compounds and gases, charts for solving mean-temperature differences, etc.

*52* • *DEWAR FLASKS.* A bulletin describing containers and flasks for liquefied gases made by Hofman Laboratories, Inc. Capacity from 5 to 1000 l. Charts show evaporation rates and other equipment for low-temperature gases.

*53* • *PROTECTIVE COATINGS.* A general catalog covering the entire line of Prufcoat Laboratories, Inc., protective coatings. Lists physical and protective qualities of all eight Prufcoat products which are protective paints of various resins for application to masonry, metal, or wood. Can be obtained to resist acids, alkalis, alcohols, oil, salts and water.

*54* • *CORROSION-RESISTANT EQUIPMENT.* An extensive catalog of all equipment made by Haveg Corp. molded from a mixture of acid-digested asbestos and synthetic resin. Bulletin gives grades of material, chemical resistance, electrical properties, standard pipes and fittings, with dimensions and illustrations, valves and pumps, fume ducts, tanks, towers, pressure and vacuum equipment, filters, accessories, agitators, agers and other equipment made by this company. In all cases specifications, description and pictures are included.

*55* • *MECHANICAL PACKINGS.* A bulletin on various kinds of packing and packing services offered by Packing Engineering Corp. The catalog is extensive and covers many types. Company differentiates between a sealing packing and a retaining packing, and describes difference. For valves, pumps, high pressure, etc. Gives installation procedures, packing rules, service recommendations for centrifugal and rotary shafts, reciprocating rods, etc.

*56* • *SPRAY NOZZLES.* Catalog from Link-Belt Co. on nonclogging spray nozzles. Installed on pipe in which a plain hole has been drilled at desired spray location. Six standard sizes. Installed by merely placing deflector over drilled hole and fastening with a U-bolt.

*57* • *FLINTKOTE DIGEST.* An industrial products digest from the Flintkote Co. which illustrates standard and specialized products for industry. Covers custom-formulated asphalt emulsions and cutbacks, asphalt rubber and resin adhesives, coatings, sealers, flooring binders, cements, sound deadener and insulating compounds, packaging specialties, laminates, etc.

*58* • *AERO HEAT EXCHANGER.* From Niagara Blower Co. a new bulletin on aero heat exchanger for cooling or controlling the temperature of a liquid or gas. Fluid to be cooled is in a closed system and the cooling is done by spray water. Heating or cooling cycles are possible and bulletin shows flow path and isometric construction view of unit.

## NEWS

(*Continued from page 52*)

### CORROSION MAJOR PROBLEM IN A.E. WORK

Jacketing uranium slugs in a nuclear reactor with a metal that will be corrosion resistant at high temperatures and that will not absorb neutrons is one of the vital problems facing the Atomic Energy Program, according to J. E. Draley of the Argonne National Laboratory, Chicago, Ill. Dr. Draley pointed out this and other problems of corrosion in the Atomic Energy Program in an address to the National Association of Corrosion Engineers last month.

The chief hazards of corrosion in the nuclear reactors, according to Dr. Draley, arise from failure of the protective covering on the uranium slugs, which would permit the escape of radioactive materials, endangering handlers and the surrounding environment, and the formation of heavy corrosion scale, thus impeding heat transfer. Other types of plants pose different problems. In the gaseous-diffusion type, for instance, efficient separation is impossible if the barrier material becomes corroded by the uranium hexafluoride. Still other difficulties arise in the chemical separation of plutonium from nuclear-reactor discharge, as the solutions used have highly corrosive tendencies.

In the nuclear reactors themselves corrosion is due chiefly to the coolant used, which may be water, air, uranium solutions, or liquid metals. As water is a desirable coolant, numerous metals have been tested for their resistance to it, among them uranium, aluminum, beryllium, zirconium, and the stainless steels and related alloys. A report on the corrosion behavior of metals in distilled water at temperatures up to 315° C. has recently been released by the Argonne National Laboratory.

Aluminum so far has proved to be the most satisfactory jacket for uranium slugs, as extensive tests, described in some detail by Dr. Draley, have demonstrated that in relatively pure water this metal is corrosion resistant up to temperatures well above the boiling point of water. At the highest temperature it behaves best in slightly acid solutions. However the pure aluminum loses its strength at elevated temperatures, and a satisfactory alloy is still to be found.

### E.M.C. URGES CONTROL OF RESERVIST RECALL

The Engineering Manpower Commission of Engineers Joint Council urged civilian control of the recall of reservists to active duty in a statement before a

(*Continued on next page*)


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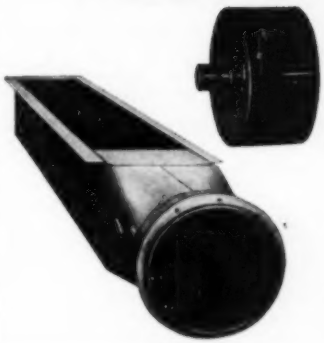

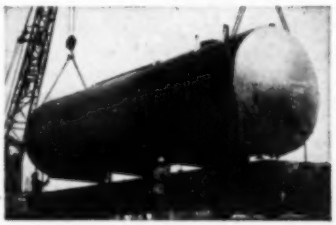

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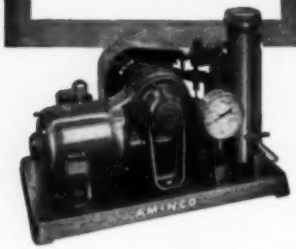

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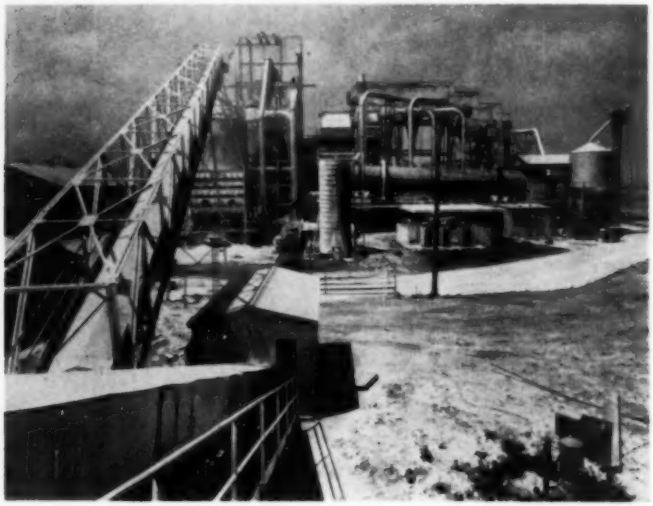
## NEW DIATOMITE PLANT AT LOMPOC, CALIFORNIA



**The largest new plant for processing diatomite to be built in the past 22 years was opened recently by the Great Lakes Carbon Corp., at Lompoc, Calif. Outdoor-type construction was used throughout, except for the final bagging operation. Conveyor on the left carries crude diatomite from the dump pits just visible in the center into the storage bin from which it is fed into the mill.**

## E.M.C. URGES CONTROL

(*Continued from page* 59)

Subcommittee of the Committee on Armed Services, U. S. Senate during hearings last month in connection with the Armed Forces Reserve Act of 1951.

In presenting the statement by Carey H. Brown, chairman, E.M.C., Norman A. Shepard, chemical director, American Cyanamid Co. and member of E.M.C. of E.J.C. and the A.I.Ch.E. stated, "There is a patriotic obligation upon every man and woman for appropriate service of which each is capable in maintaining the security of the United States. We are faced with a critical shortage of engineers and scientists." There is a current backlog of demand for more than 60,000 engineers. In addition it has been reliably estimated that we need 30,000 new engineers each year. For the next five years the colleges will be graduating an average of 19,000.

In stressing the control of a civilian agency, Dr. Shepard asserted:

> Since this selection will be for service in the essential economy as well as for military service, reservists other than those in active organized units of the Ready Reserve should be called to active duty by a civilian agency such as Selective Service.

In maintaining the needed flow of young engineers, Dr. Shepard stated that the recall to active duty of the required number of professional and scientific students should be held in abeyance until completion of their professional study.

## POWER PLANT FOR IDAHO

A Bureau of Reclamation contract for construction of the Palisades Dam and power plant on the South Fork of the Snake River about 50 miles from Idaho Falls, Idaho, has been awarded to the J. A. Jones Construction Co., Seattle, Wash., and the Charles H. Tompkins Co., Washington, D. C., on a joint-venture low bid of $29,180,346.

Under the contract, the project which will turn out 700 million kw.hr. of hydroelectric power annually must produce power in 1,700 days.

Earlier last year, need for the project was emphasized by the Defense Electric Power Administration, which pointed out that the development of phosphate for defense needs, as well as power for the atomic energy installations at Arco, Idaho, was dependent upon the availability of hydroelectric power from the Palisades Project.

Palisades Dam, a 260-ft. high 2,000-ft. long structure—the largest earthfill structure yet built by the Bureau of Reclamation—will provide a 1,400,000 acre-foot storage reservoir, furnishing supplemental water for 650,000 acres of irrigated farmland in the Snake River Valley and water to drive the three 39,500 hp. turbines to be installed in the three-generator, 114,000 kw. power plant.

# LOCAL SECTION

## OHIO VALLEY

This Section held a dinner meeting at the Engineering Society headquarters in Cincinnati, May 5. At the business meeting the following officers were elected for next year:

*Chairman*.....Arthur C. Greber
*Vice-Chairman*.....T. B. Wiehe
*Secretary*.....Stanley N. Baechle
*Treasurer*.....Robert C. St. John

This was the annual student night meeting at which two students from the chemical engineering department of the University of Cincinnati presented papers. The first paper, "Titanium and Its Alloys," was by William P. Koster, and the second, "The Salting Out Effect in Separation Operations," by Don D. Schiewetz.

*Reported by N. W. Morley*

## BOSTON

On April 25, 1952, a regular meeting of the Section at the M.I.T. Graduate House heard Joseph W. Wulfeck of Tufts College psychology department speak on "Human Engineering." The problem of tailoring the machine to the human factor or vice versa, provoked much discussion.

The regular annual election of officers of the Ichthyologists for 1952-53 resulted in the following slate:

*Kingfish (Chairman)*—J. Edward Vivian, M.I.T.
*Mackerel (Vice-Chairman)*—Randolph Antonsen, Godfrey L. Cabot, Inc.
*Smelt (Secretary)*—Arthur G. Smith, Monsanto Chemical Co.
*Shark (Treasurer)*—Craig Angell, Artisan Metals Co.

The Second Annual Technical Symposium covering "Economic Aspects of Chemical Engineering Projects" held April 16, 1952, at the University Club in Boston, was highly successful. Approximately 260 chemical engineers attended the afternoon sessions and dinner.

*Reported by G. J. Odom*

## AKRON

Recently elected officers are as follows:

*Chairman*—John W. Kosko, Goodyear Tire & Rubber Co., Akron, Ohio
*Vice-Chairman*—Archie B. Japs, B. F. Goodrich Research Center
*Secretary*—Warren E. Phillips, Firestone Tire & Rubber Co.
*Treasurer*—H. H. Baker, Jr., White Industrial Sales & Equipment Co.

*Reported by J. W. Kosko*

(*More Local Section on page 62*)

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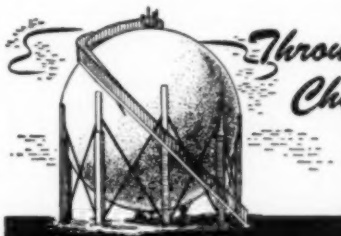

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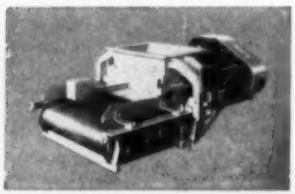

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AMERICAN CANCER SOCIETY


## LOCAL SECTION NEWS

(*Continued from page 61*)

### NORTHEASTERN NEW YORK CHEMICAL ENGINEERS' CLUB

The sixth meeting of the club was held in Schenectady March 20. The 28 persons present reviewed the bylaws and accepted them as amended with a few minor changes. Movies on "Bubble Cap Performance," "Curves of Color," and "Resistance Welding" were shown.

The club met for the seventh meeting April 17 in the auditorium of the G. E. Research Laboratory, Schenectady, N. Y. The guest speaker at the meeting was Thomas H. Chilton of Du Pont Co.; his topic was "Why an Engineering Society?" The meeting was preceded by a dinner in the Laboratory cafeteria and was attended by 59 members.

*Reported by Roger E. Larson*

### SOUTH TEXAS

This Section had a field trip through the Eastern States Petroleum Co. refinery in Houston, Tex., May 28, 1952, where an inspection was made of the platforming plant for the manufacture of aromatics and the Udex plant, which purifies the product. The social hour, dinner and meeting which followed were held at the Blue Room of the Alabama Cafeteria. The speaker of the evening was J. A. Gerster, professor of chemical engineering, University of Delaware. Dr. Gerster's talk was titled "Mass Transfer Rates on Bubble Plates."

A previous meeting consisted of a trip through the Southwest Research Institute in San Antonio and a talk by Harold Vagtborg on "Progress through Inventions."

*Reported by W. R. Trutna*

### WESTERN NEW YORK

At the April 10 dinner meeting, held at the Hotel Stuyvesant, Buffalo, N. Y., the 1952-53 officers were elected as follows:

*Chairman*.....J. E. Troyan
*Vice-Chairman*.....J. A. Bergantz
*Secretary-Treas*.....J. W. Casten
*Directors:* E. C. Mirus, W. C. Greenwald, H. M. Tatomer
*Delegate to Tech. Socs. Council*—H. A. Coit

The speaker of the evening was Stephen A. Laurich, design engineer for the Struthers-Wells Corp., who gave a talk on "Industrial Crystallization." Mr. Laurich traced the beginning of crystallization to early times when solar evaporation was used in producing salt. In more recent times, he claimed, crystallization was carried out in cooled cascade tanks; later in tanks equipped with cooling coils and agitation, and finally in the present industrial crystallizer which makes use of a crystal classification and

growing section and high circulating rates through a heat exchanger. In present times, crystallization has become an important unit operation and large tonnages of organic and inorganic crystals are produced.

Sixty were in attendance at the dinner and about sixty-five at the meeting.

*Reported by W. C. Greenwald*

## CHARLESTON

The sixth meeting of the year was held April 15 at the Charleston Recreation Center. A total of 105 members and guests were present.

The program portion of the meeting consisted of a discussion by a three-man panel of the proper applications of the various types of stainless steel and the ways in which the most effective use of stainless alloys can be made during the current period of shortages. Barclay Morrison, manager of process tube sales development, Alloy Tube Division, The Carpenter Steel Co., Union, N. J., outlined the developments leading up to the present supply situation and then discussed the fundamentals of corrosion resistance in stainless steel. He was followed by S. E. Doughty, metallurgist, Alloy Tube Division, The Carpenter Steel Co., and J. J. Folz, engineer in charge of stainless steels, Horace T. Potts Co., Philadelphia, Pa. Questions on such subjects as welding and the overcoming of troubles arising from it, stress corrosion, the effects of cold working, and availability of material were answered by the members of the panel.

*Reported by G. K. S. Connolly*

## COASTAL GEORGIA CHEMICAL ENGINEERS CLUB

Members of the Club met April 4 to hear R. S. Ingols, research professor, Georgia Institute of Technology Experiment Station, talk on "Water Problems in Industry." The meeting was held at the Mary Calder Golf Club, Savannah.

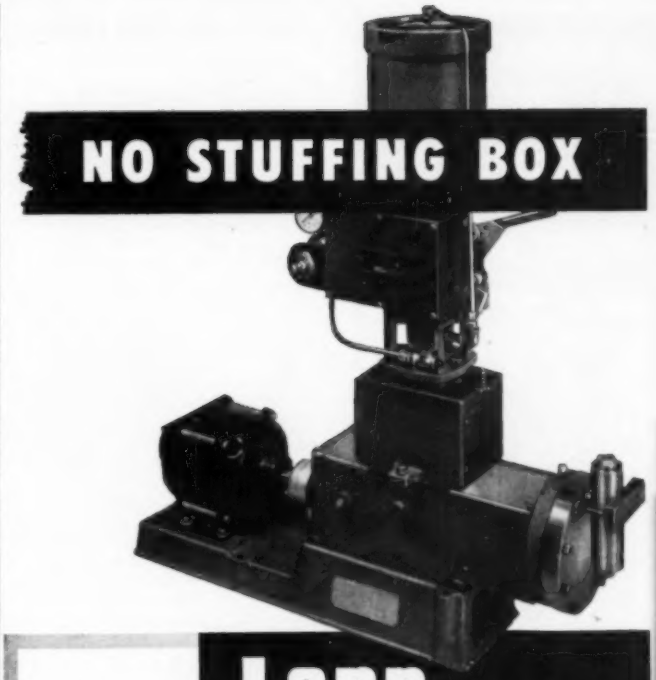
An informal discussion was held with W. I. Burt, President of A.I.Ch.E. and vice-president, manufacturing, the B. F. Goodrich Chemical Co., March 28, at the Oglethorpe Hotel in Brunswick. He talked on Institute activities.

## DETROIT

John N. Cadaret of Wayne University addressed this Section on "Management Myths" at the April meeting. Approximately 75 members attended the nontechnical session. Present business practices, labor relations, wage administration and many other fields of business were discussed.

*Reported by Dwight Miller*

(*More Local Section on page 64*)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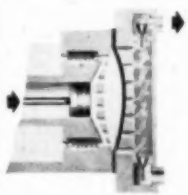
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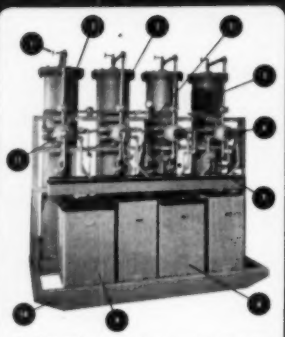

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## LOCAL SECTION NEWS

(*Continued from page 63*)

### CENTRAL OHIO

Messrs. Mueller, Peterseim and Sachsel, Battelle Memorial Institute, cooperated with members of the local American Electroplaters Society Chapter in the presentation of a half-hour TV program on April 27 discussing tires, paint, and trim of automobiles in a series entitled "Nation on Wheels," produced under the auspices of the Columbus Technical Council. The latter organization was created a number of years ago to promote communications among the various local sections of national technical societies and promote good public relations for the engineering professions represented by these societies.

This Section on April 30, 1952, heard Kenneth Cottingham, chief geologist of the Ohio Fuel Gas Co., discuss "Underground Storage of Natural Gas." The speaker described how his company had developed large inventories of natural gas by storage at about 1000 lb./sq.in. in spent gas wells throughout Ohio, thus assuring an adequate supply of natural gas despite fluctuating demands.

*Reported by George F. Sachsel*

### NORTHERN CALIFORNIA

On April 21, 1952, this Section met jointly with the Student Chapter at Stanford University. The event, which was arranged by the students under the chairmanship of I. du P. May, president of the Student Chapter, began with dinner at Ferranti's near Palo Alto. Later the 110 persons attending adjourned to the Stanford campus.

The program began with the presentation of the Student Chapter Awards for the year. These annual awards, each consisting of one year's Junior membership in the National A.I.Ch.E. and a Junior key, are given by the Section to the two individuals chosen by the committee as the most outstanding members of the Student Chapters in Northern California. The Award for the University of California went to Edward L. Scriven of Burlingame, and that for Stanford University to Robert M. Kendall of Pasadena.

E. W. Costich, Mixing Equipment Co., presented a talk illustrated by motion pictures showing the results of research into the "Principles of Mixing." Following this, the group toured the laboratories and facilities of the University under the direction of C. G. Lindquist, head of the department of chemical engineering. The various graduate projects were described and explained by the students participating in the work.

*Reported by D. F. Rynning*

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## NEW YORK

Officers for 1952-53 elected at the May 20 meeting are as follows:

*Chairman*.....Howard L. Malakoff
*Vice-Chairman*.....Francis B. White
*Secretary*.....Crawford Bown
*Treasurer*.....Max Strawn
*Member-At-Large*.....E. L. Demarest

*Reported by C. H. Chilton*

## EL DORADO

Fifty-six members and guests heard Maurice E. Barker at the Garrett Hotel, April 25, discuss "Activated Charcoal; Its History and Some of Its Uses." Dr. Barker is with the department of chemical engineering, University of Arkansas, Fayetteville, Ark.

The month previous 65 members and guests at Dewey's Steak House were present at a round-table discussion on, "Sulfur Recovery from Sour Gases," conducted by J. S. Gilliam, of Mathieson Chemical Corp. and some of his coworkers. He substituted for J. L. Parker, of the Girdler Corp., the scheduled speaker.

At other meetings during the current year Kenneth Barnes, Columbian Carbon Co., spoke on "The Development and Manufacture of Oil-Type Furnace Carbon Black," and Horace W. Lee, of Food Machinery and Chemical Corp., on "The Development of Insecticides for three Decades."

*Reported by A. A. Feerick*

## ROCHESTER

The sixth meeting of the 1951-1952 season was the annual plant trip held at the Pfaudler Co. on April 16 when 140 members toured the plant. Luncheon was served by the host. This was followed by the tour and a Seminar on the latest developments in glass lining, agitation, and accessories.

*Reported by Irving Siller*

## LOUISVILLE

At the meeting of this Section held at Speed Scientific School Auditorium, May 13, Warren K. Eister, chemical technology division, Oak Ridge National Laboratory, which is operated by Carbide and Carbon Chemicals Corp., spoke on "The Engineer in The Atomic Energy Industry." The newly elected officers for the next year are as follows:

*Chairman*.....Hans P. Hansen
*Vice-Chairman*...Wilmer Reynolds, Jr.
*Sec'y-Treas.*.....Hans J. Treumann
*Exec. Comm.*: John L. Nelson
John C. Weyrich

*Reported by J. Schaaf*


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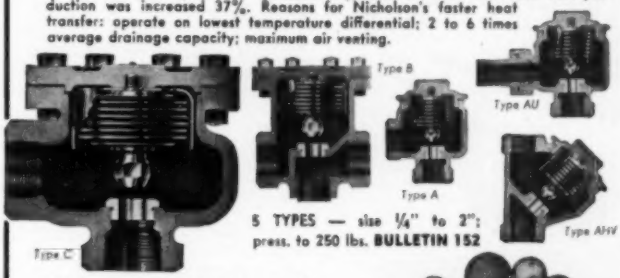


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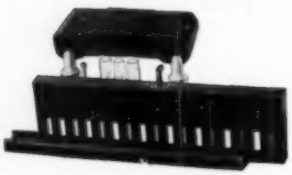

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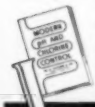

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# PEOPLE

**D. H. Gordon** has recently changed positions. He is now president and general manager of Industrial Machine Works, Inc., Waynesboro, Va., which is engaged in machining, manufacturing, engineering and design, chiefly of mechanical items. His former position was with DuPont Co., Waynesboro, Va., as research supervisor in the technical division of the textile fibers department. He received his B.S. and M.S. degrees and also a Ph.D. from the University of Wisconsin.

**R. P. Dinsmore,** vice-president in charge of research and development of the Goodyear Tire & Rubber Co., Akron, Ohio, recently delivered the Seventh Foundation Lecture of the Institution of the Rubber Industry in Wolverhampton, England, titled "Economic and Physical Aspects of GR-S Modifications." Mr. Dinsmore joined Goodyear in its experimental department in 1914, upon his graduation from Massachusetts Institute of Technology. He has held various positions, including that of chief chemist, and was appointed to his present position in 1943.

**Ralph J. Holtschlag** has been appointed an operating superintendent at Monsanto Chemical Co.'s John F. Queeny plant, St. Louis, Mo. He has been group leader in charge of interim manufacturing facilities for the company's organic chemicals division. Upon graduation from St. Louis University, in 1938 he joined Monsanto as an analyst, later becoming a research chemist and group leader.

**Joe Cresce,** who for the past three months has been acting plant manager of Monsanto Chemical Co.'s Nitro (W. Va.) plant, was recently named plant manager. He has been assistant plant manager since 1947, and has been with Monsanto at Nitro since 1936 when he was graduated from West Virginia University with a B.S. degree in chemical engineering.

**B. P. Martinez** has recently been named chief of the U. S. Bureau of Mines Stream Pollution Research Station for the Northeastern Region at College Park, Md., where all activities of the section in the 17 Northeastern states will be consolidated. Prior to accepting this new position, Mr. Martinez was process engineer for a steel firm at Sparrows Point, Md. He is a chemical engineering graduate of the Massachusetts Institute of Technology.

## FRED OLSEN HEADS IND. RES. INST.

Fred Olsen, vice-president for research and development, Olin Industries, Inc., East Alton, Ill., has been elected president of the Industrial Research Institute, Inc., New York, N. Y. Dr. Olsen is also an Olin director and has been director of the company's research and development. He became associated with Olin Industries in 1929 and has done considerable work in connection with cellulose and high explosives.

New members of the Institute board elected for three years are: **L. B. Hitchcock,** president, National Research Laboratory, Inc., Oakdale, L. I., N. Y.; and **H. G. Vesper,** president, California Research Corp., San Francisco, Calif.

**Robert T. Wallace,** superintendent of the coating resin and alkyd molding compound plants at Plaskon division of Libbey-Owens-Ford Glass Co., Toledo, Ohio, has recently been awarded one of the 14 annual Sloan fellowships for executive development at Massachusetts Institute of Technology. A graduate of the University of Michigan with a B.S. in chemical engineering, he also took selected graduate courses at Illinois Institute of Technology. Before going to Toledo, he worked for Eastman Kodak Co., at Rochester, N. Y., and Carbide & Carbon Chemicals Co., Whiting, Ind.

**Roy Newton,** vice-president of Swift and Co., was presented the 1952 Medal of the Industrial Research Institute, "awarded for outstanding accomplishments in the management field of industrial research." The medal was presented to Dr. Newton for pioneering in long-range research on food products and for his leading industry to high standards of public service.

**David M. Shapleigh** has recently been appointed sales engineer in the Zenith division of Jackson & Church Co., Saginaw, Mich. Associated with technical sales and service in the paper and pulp and textile industries for 20 years, Shapleigh was former manager of Superba Mills, Inc., Hawkinsville, Ga. He formerly held positions with Paper and Industrial Appliances, Inc., New York, Hercules Powder Co., Wilmington, Del., Brown Co., Berlin, N. H., and Amoskeag Mfg. Co., Manchester, N. H. He received his degree in chemical engineering from the University of Maine.

**G. V. Slottman** has recently been elected vice-president of Air Reduction Co., Inc., New York. He has been the company's director of research and engineering since 1949 and will continue in charge of that activity. Dr. Slottman is a graduate of the Massachusetts Institute of Technology. He received his Ph.D. from the University of Berlin and returned to MIT as a professor of chemical engineering where he remained until 1930 when he went with United States Steel Companies, Ltd., Sheffield, England, as chief combustion engineer and iron works manager. He rejoined Airco in 1934, and subsequently has served as manager of the general technical sales department, technical assistant to the vice-president and director of research and engineering.

**Forrest O. Calhoon,** president of the Disco Co., division of the Pittsburgh Consolidation Coal Co., became affiliated with Koppers Company, Inc., Pittsburgh, Pa., effective May 1. Mr. Calhoon, who was associated with Koppers from 1927 to 1946, will serve as a technical adviser to Fred C. Foy, vice-president and general manager of the tar products division. At the time he left Koppers Mr. Calhoon was general operating superintendent of the tar products division. In 1948 he joined the Disco Co. as assistant to that firm's president, was made vice-president in 1950 and assumed the position of president in 1951. For two years prior to joining Disco, Mr. Calhoon was plant manager of the Varcum Chemical Corp., Niagara Falls, N. Y.

**Herman M. Zabel** has recently been appointed executive vice-president of Roger Williams, Inc., N. Y., engineering and economic consultants to the chemical process industries. Prior to his present position, Mr. Zabel was director of research and development of Chemical Enterprises, Inc., and before that, associate editor of Chemical Week and engineering editor of Chemical Industries. After graduation from Kansas State College and Columbia University he was associated with Du Pont Co. and General Aniline and Film Corp.

*(More About People on page 68)*


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## PEOPLE

(*Continued from page 67*)

**Harrison C. Blankmeyer** was recently appointed scientific director of the Fiberglass Research and Development Laboratories of Owens-Corning Fiberglas Corp., Toledo, Ohio. He will assist the vice-president in coordinating product development and process improvement programs. A 1936 chemical engineering graduate of the University of Illinois, from 1937 to 1951, while he was employed by Joseph E. Seagram & Sons in Louisville, he served as a technical engineer, director of education, plant manager, technical director, services manager, and assistant to the vice-president. From early 1951 up until his recent appointment, he was assistant to the president of Kingan & Co., Indianapolis, meat-packing firm.

### MORGEN WITH NAT. SCIENCE FOUNDATION

Dr. Ralph A. Morgen, director of the Engineering and Industrial Experiment Station of the University of Florida, has been appointed to the staff of the Division of Mathematical, Physical and Engineering Sciences of the National Science Foundation. Dr. Morgen began his new duties June 1, 1952, when he took charge of the Foundation's research support program in the engineering sciences.

Formerly consulting chemical engineer to various government and private research and development agencies, Dr. Morgen joined the faculty of the University of Florida in 1938. Before that he was manager of research and development for Black, Sivalls and Bryson Manufacturing Co., Kansas City, Mo. During World War II Dr. Morgen served as consultant with the Office of Scientific Research and Development.

### CORRECTION

In an item on Ernest W. Reid's election to the board of directors of Commercial Solvents Corp., it was erroneously stated in the April issue of "C. E. P." page 79, that immediately prior to his going with Commercial Solvents he was associated with the War Production Board. Dr. Reid is president of Corn Products Refining Co., New York. The fact is that it was immediately prior to his going with Corn Products Refining Co. that he was associated with the War Production Board.

**A. I. Johnson** has been appointed to the professional staff, department of chemical engineering, University of Toronto, Canada. He is a graduate of that department, University of Toronto (1946). He obtained his M.Ch.E. degree from Brooklyn Polytechnic Institute in 1948 and the Ph.D. degree from Toronto in 1950. For the past two years he has been assistant professor of chemical engineering at Johns Hopkins University, Baltimore, Md. Dr. Johnson was the holder of many scholarships as an undergraduate and graduate student, including the Wallberg Research Fellowship, and has specialized in his graduate work in the field of unit operations.

**Richard C. Wood** has been named assistant to the vice-president in charge of product divisions of Commercial Solvents Corp., New York. For the past 16 years, Mr. Wood has held executive and development positions in the chemicals industry and previously with General Aniline and Film Corp. He received his B.S. in chemical engineering from the University of Rhode Island and his Doctorate from New York University. During World War II he served overseas with the Army Air Force.

**Robert M. Cornforth** has recently been appointed director of foreign sales of Houdry Process Corp., Philadelphia, Pa. He joined the corporation in 1945 as a sales engineer and was made manager of sales in 1948. Prior to his association with Houdry he was a chemical engineer with Standard Oil Company of Indiana. During World War II, he was assigned by the Army Corps of Engineers to the Petroleum Administration for War and also served as Executive Secretary of the Government's Aviation Gasoline Committee. He received a B.S. degree in chemical engineering from the University of Tennessee, an M.S. from M.I.T., and an M.B.A. degree from Northwestern University.

## WEINRICH ADVANCED BY CATALYTIC

Whitney Weinrich has been appointed manager of the process engineering section of the Catalytic Construction Co., Philadelphia, Pa. In this capacity he will direct all research and development, pilot plant investigation and process design for the oil refinery, chemical, and atomic energy projects of the company. In 1950 he went with Catalytic Construction to direct the process development for an uranium ore refinery under Catalytic's contract with the New York Operations Office of A.E.C.

A graduate of Massachusetts Institute of Technology with a Masters degree in chemical engineering Mr. Weinrich has been identified with the Gulf Research and Development Co. for ten years, and later served as process engineer with the Petroleum Administration for War. He has been associated with Phillips Petroleum Co., in Bartlesville and Houdry Process Corp., in Marcus Hook. Weinrich is the holder of 15 issued patents. He taught chemical engineering courses at the University of Pittsburgh, Pennsylvania, and Delaware.

**Ernst A. Hauser,** professor of chemical engineering at M.I.T., will conduct a special program in elastic high polymers as part of the summer program in the colloid chemistry of Elastic High Polymers in Science and Industry to be held at the Institute from June 16 to July 5.

**Ralph E. Werley, Jr.,** has joined Jefferson Chemical Co., New York, as a representative on its sales department staff. Mr. Werley, who received his B.S. in chemical engineering from Lehigh University in 1948, was until recently associated with Cities Service Oil Co.

**Herbert E. Silcox** has been appointed plant engineer and assistant plant manager at the Stonewall plant of Merck & Co., Inc. He will assume his new position about July 1, meanwhile spending part of his time in Elkton, Va. He began with Merck in 1942 as a chemical engineer in the process development department. He was appointed assistant director of the developmental research department in 1947 and in 1950 was made director. Dr. Silcox received his B.S. and M.S. degrees in chemistry at the University of New Hampshire, and his Ph.D. in chemical engineering at the University of Illinois.

**Thomas K. Sherwood,** dean of engineering at Massachusetts Institute of Technology, Cambridge, Mass., has asked to be relieved of his administrative duties in order to devote full time to teaching and research as professor of chemical engineering. He will be succeeded by Vice-Admiral E. L. Cochrane, U.S.N. (retired), currently on leave from his post as head of the department of Naval Architecture and Marine Eng. at M.I.T., serving in Washington as head of the Federal Maritime Board.

**V. C. Williams,** formerly associate director of research, Central Research Laboratories, General Aniline and Film Corp., Easton, Pa., is now with Mississippi River Fuel Corp., St. Louis, Mo., as director of chemical development.

(*More About People on page 71*)


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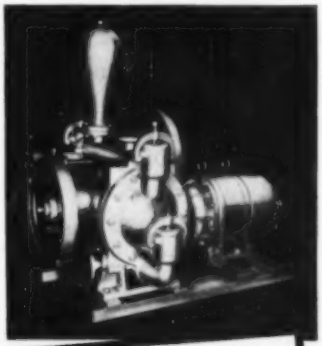

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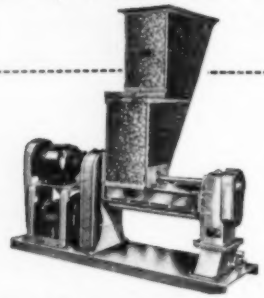

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**CHEMICAL ENGINEER**—B.S. Missouri School of Mines. Three years' domestic and foreign experience in petroleum (reservoir) engineering. Single. Not yet U. S. citizen. Desire position in South America. Presently located in Venezuela. Box 8-6.

**REGISTERED PROFESSIONAL ENGINEER**—M.Ch.E. Four years' process design, three years' mechanical design experience. Available for part-time work evenings and/or Saturdays at home or Metropolitan New York. Box 9-6.

**CHEMICAL ENGINEER**—M.Ch.E. Four years' experience with organic and petrochemicals in process engineering and development, plant studies, trouble shooting, economic studies, pilot plant and commercial plant design. Desire challenging new position with future. Box 10-6.

**CHEMICAL ENGINEER** — Ph.D. Age 31, family. Nine years teaching, research and industrial organic chemical development experience. Desire academic or industrial research and development position. Box 11-6.

**CHEMICAL ENGINEER**—Fifteen years directing and performing production, design, and pilot plant work for food and organic chemical industries. Desire supervisory position in technical administration, production or engineering. Employed. Major developments. Publications. Box 12-6.

**CHEMICAL ENGINEER**—B.S. Age 32, family, veteran. Four years' diversified laboratory and refinery chemical experience, major oil company. Laboratory managment, refinery treating problems etc. Prefer West. Oil or related industry with opportunity to advance. Box 13-6.

**CHEMICAL ENGINEER**—M.Ch.E. 28. Three years' experience with leading instrument manufacturer in technical writing, technical service, and sales. Desire challenging position in development or production, requiring experience in instrumentation. Cleveland area preferred, others considered. Box 14-6.

**MECHANICAL ENGINEER** — B.S.M.E., A.S.M.E., A.I.Ch.E., registered, married, veteran. Ten years' experience, majority rayon industry economics and design. Desire permanent responsible advertising, sales, production position. Prefer Philadelphia or Southeast U. S. Required salary $9000. Please reply Box 15-6.

## PEOPLE

(*Continued from page 69*)

**Robert E. Workman,** formerly St. Louis district manager of Goodyear Tire & Rubber Co.'s chemical division, has been named to the newly established post of manager of commercial development, with headquarters in Akron, Ohio. Workman joined Goodyear in 1942 as a factory office trainee in Akron. The following year he was named junior research chemist and in 1946 became senior chemical engineer. In the research department, his work was in the fields of rubber, plastics and protective coatings, in which fields he has several patents and a number of publications. Early in 1947 he was appointed special representative in Akron, and the next year transferred to St. Louis in the same capacity. He has been chemical division district manager there since April, 1951.

**Jose B. Calva,** chemical engineer, was recently presented with the Leo Altenberg award for his work in the field of fur technology. Dr. Calva is the inventor of the Calva process for making imitation fine furs from sheepskins by permanently straightening wool fibers chemically.

## *Necrology*

### A. B. NEWMAN

Albert Broadus Newman, President of the American Institute of Chemical Engineers (1948), and professor of chemical engineering and former dean of engineering at the College of the City of New York, died May 9, at the age of 64.

Dr. Newman was active in Institute affairs throughout the 37 years of his membership. He had served as a Director for the period 1936, 1937, and 1938; and again in 1940, 1941 and 1942; as Vice-President in 1947 and as President in 1948. He also served on many of its committees, particularly as a member and later as chairman of the Chemical Engineering Education and Accrediting Committee.

A native of Toronto, Dr. Newman received a B.A. in 1910 from the University of Michigan and a doctorate in 1928. His first teaching position was as an instructor at Pennsylvania State College and subsequently he engaged in private industry. In 1927-28 he was professor of chemical engineering and in charge of the department at Cooper

(*Continued on page 72*)

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C-11


## NECROLOGY

(*Continued from page 71*)

Union and for the next ten years was professor of chemical engineering and department chairman at City College. In 1938 he was appointed dean of the School of Technology at City College. He also lectured in chemical engineering at New York University's graduate division during 1941-47 and during that period served as consultant for Northwestern University. During 1945-46 he took leave of his teaching duties at City College to represent the Government in negotiations with representatives of the British, French and Soviet governments. Dr. Newman was a member of many professional and technical societies.

### D. A. PRITCHARD

David A. Pritchard, a native of Wales, came to the United States in 1910 and was associated with the Pennsylvania Salt Manufacturing Co., and Du Pont's Philadelphia Works. Subsequently he returned to England becoming affiliated with Imperial Chemical Industries, Ltd., and remaining until 1922 when he went to the Canadian Salt Co., at Windsor, Ont. Before his retirement in 1946 he had been production manager of the heavy chemicals division of Canadian Industries Ltd. and was technical manager of the chemical group. He was a pioneer in the alkali industry. Mr. Pritchard received his academic and technical education in England.

*Chemical Engineering Progress* has recently received news of the deaths of the following:

**William Lee Judefind,** manager of process control, Davison Chemical Corp., Baltimore, Md.

**H. D. Batchelor,** director of research, National Carbon Co., Cleveland, Ohio.

**Alex Newhouse,** process engineer, Dayton Rubber Co., Waynesville, N. C.

**Alvin C. Goetz,** mgr., technical service department, The Eagle-Picher Co., Cincinnati, Ohio.


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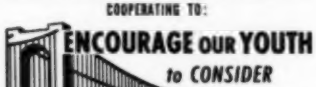


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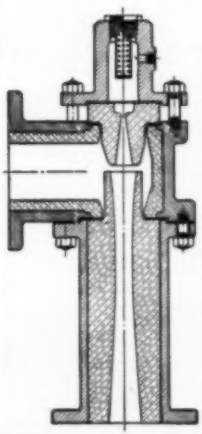

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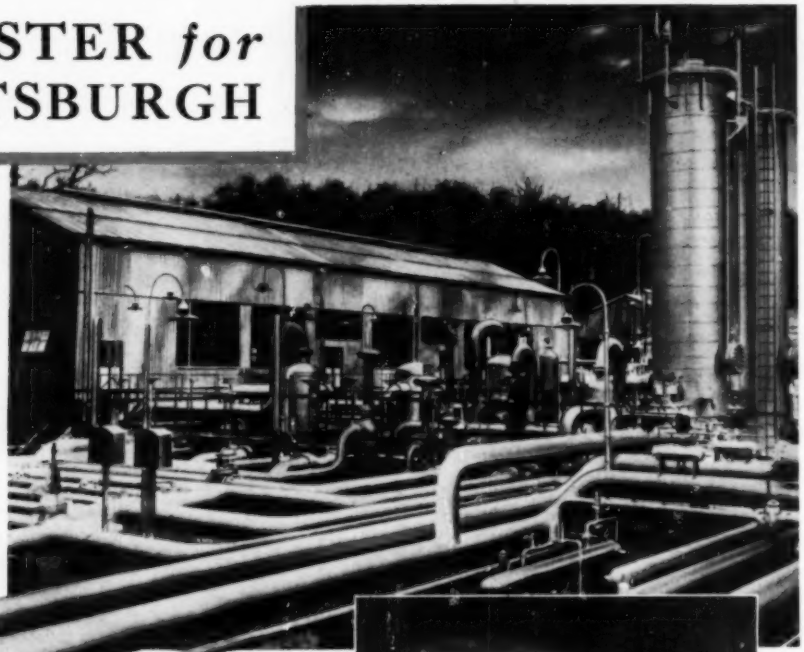

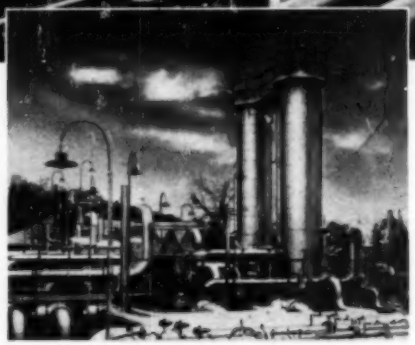

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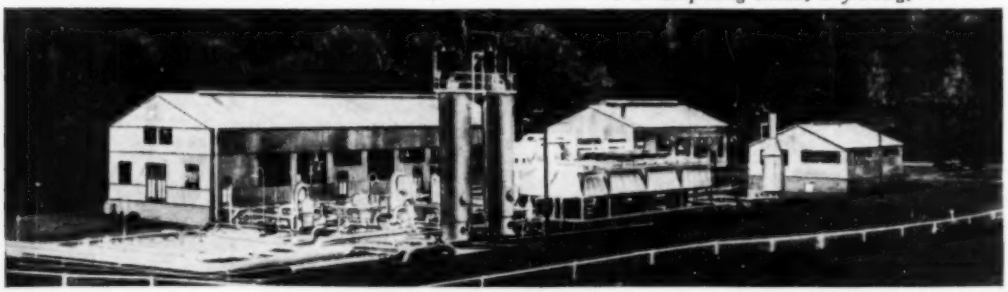

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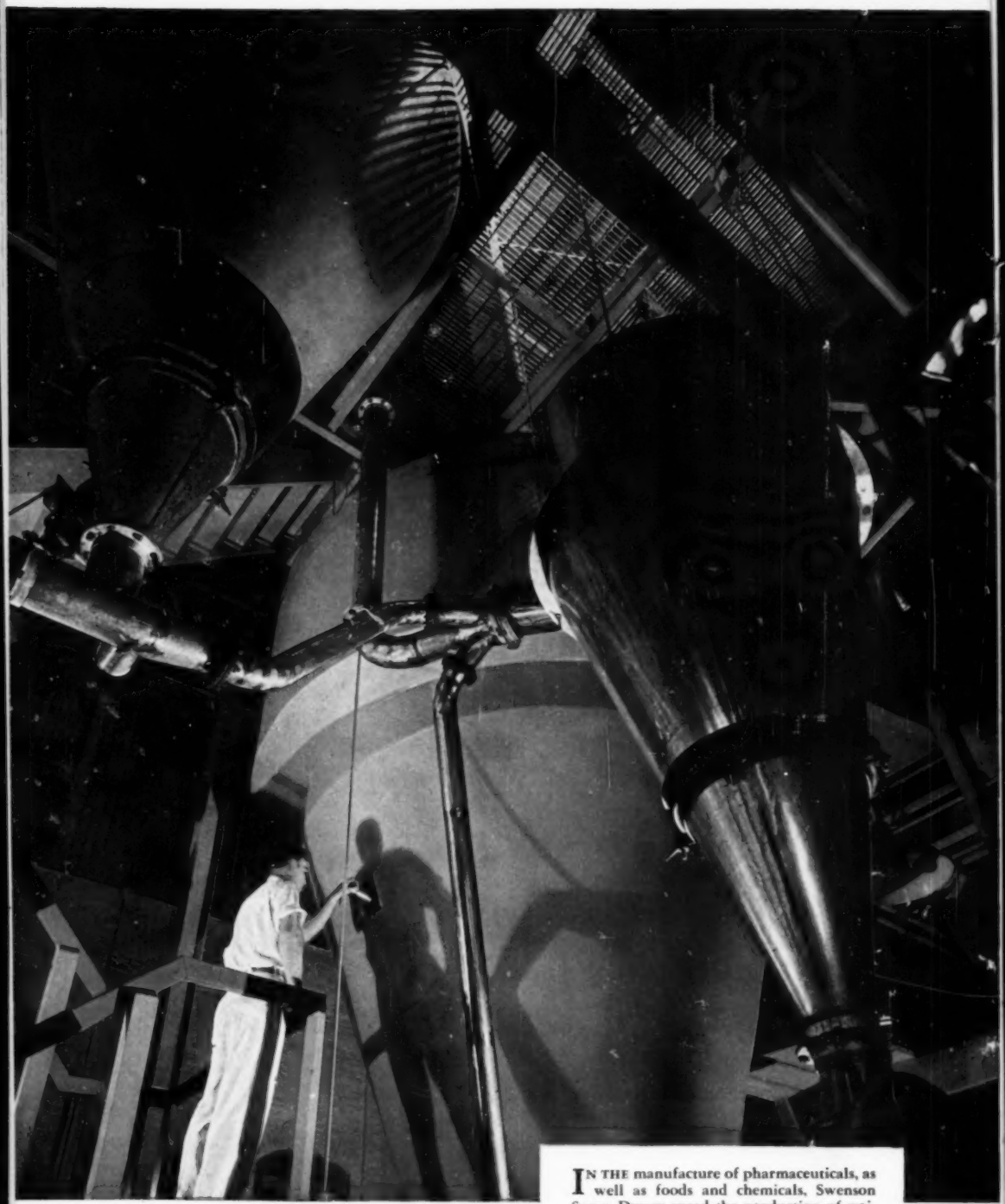

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